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U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

PROCEEDINGS

OF THE

THIRTEENTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD, AT

WASHINGTON, D. C.,

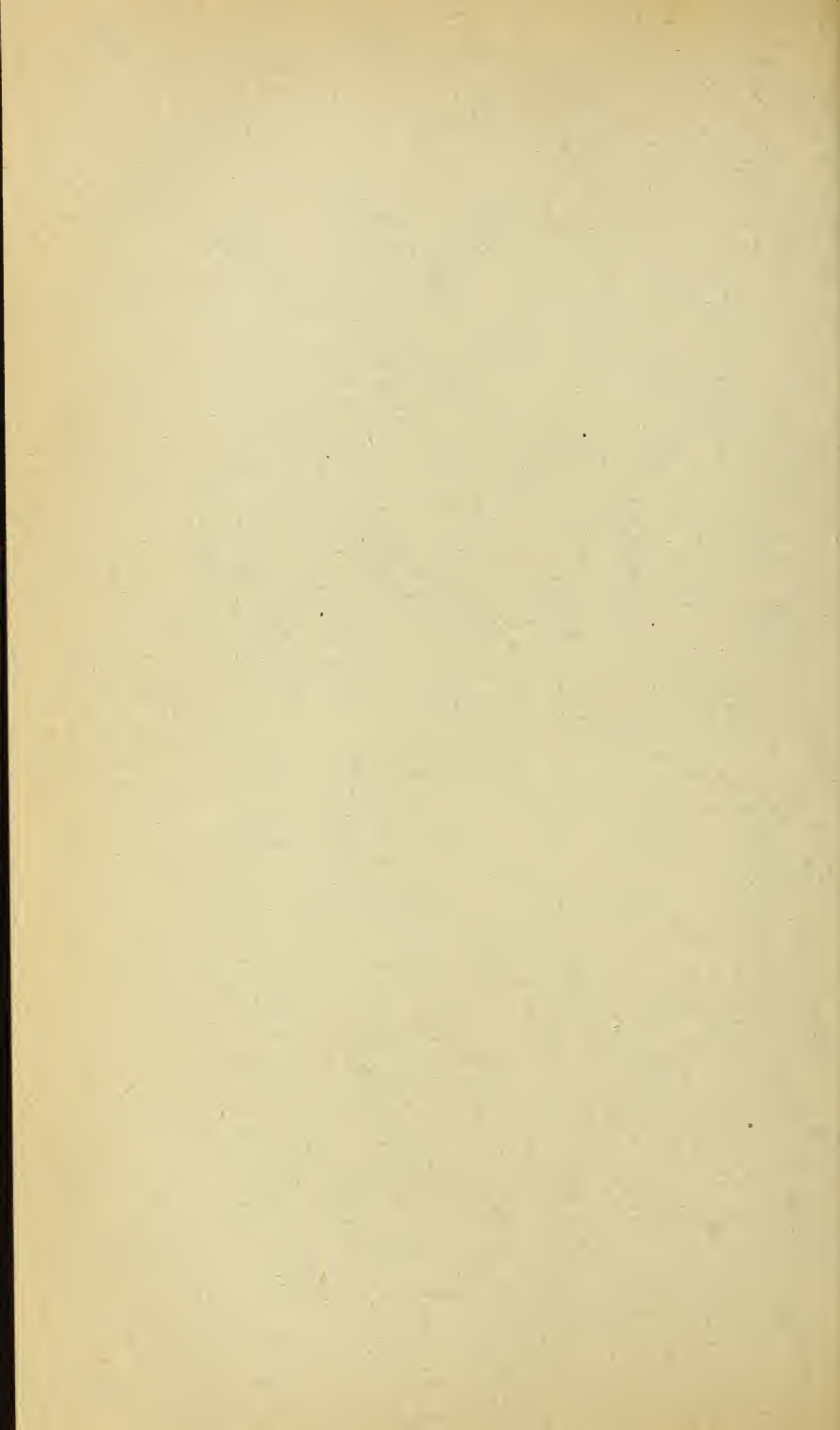
NOVEMBER 6, 7, AND 9, 1896.

EDITED BY

HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.



WASHINGTON:
GOVERNMENT PRINTING OFFICE.
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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., December 16, 1896.

SIR: I beg to transmit herewith, for your inspection and approval, the manuscript of the proceedings of the thirteenth annual meeting of the Association of Official Agricultural Chemists, and ask that it be printed as Bulletin No. 49 of the Division of Chemistry.

H. W. WILEY,
*Chief of the Division of Chemistry and Secretary of
the Association of Official Agricultural Chemists.*

Hon. J. STERLING MORTON,
Secretary of Agriculture.

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PROCEEDINGS OF THE THIRTEENTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICUL- TURAL CHEMISTS.

FIRST DAY.

FRIDAY—MORNING SESSION.

Pursuant to a call issued by the executive committee, the Association of Official Agricultural Chemists met at 10.30 a. m., Friday, November 6, in the lecture room of the National Museum.

The president, Mr. B. B. Ross, called the convention to order and nominated the following committee on order of business, viz: L. L. Van Slyke, G. L. Teller, and H. J. Patterson. The committee, after consultation, reported the following order of business:

First. President's address.

Second. Report on methods of determining nitrogen, and reading of papers relating thereto.

Third. Report on methods of analyzing potash, and reading of papers relating thereto.

Fourth. Report on methods of soil analysis, and reading of papers relating thereto.

Fifth. Report on methods for the analysis of phosphoric acid, and reading of papers relating thereto.

Sixth. Report on methods of analyzing foods and feeding stuffs, and reading of papers relating thereto.

Seventh. Report on methods of analyzing carbohydrates; papers and discussion.

Eighth. Report on methods of analyzing fermented and distilled liquors; papers and discussion.

Ninth. Report on methods of analyzing tanning materials; papers and discussion.

The election of officers for the ensuing year was fixed as a special order for Saturday afternoon, November 7, at 3 o'clock.

The president read the annual address to the association, which follows.

ADDRESS OF THE PRESIDENT.

GENTLEMEN OF THE ASSOCIATION: Our organization assembles to-day in its thirteenth annual convention after the lapse of a longer period than has intervened heretofore between any two consecutive meetings. The date selected for this meeting by vote of the executive committee was determined upon in order to enable chemists connected with the work of experiment stations and agricultural colleges to attend the meetings of this association and also the sessions of the convention of agricultural investigators and educators which assembles in this city on the 10th instant, and I trust that we have present among us some members who have been unable heretofore to attend regularly the meetings of this body.

The long interim between the last convention and the present has no doubt afforded to the reporters, and those taking part in the work of cooperative and individual investigation, additional opportunities for practical tests and studies of our methods of analysis, and I trust that the reports and results presented for our consideration will make easy the task of passing upon the questions awaiting the action of this convention.

The work of our association has become so comprehensive in its scope and so diversified in its character that it is impossible in the short time at my disposal to touch even in a cursory manner upon the varied subjects and questions to be considered by this body.

The various departments and divisions of the work coming within the province of the association is, moreover, so well covered by committees and reporters that it would be an act of supererogation on my part to enter into the detailed consideration of any individual topic or question which might demand our attention, nor will I anticipate the presentation of the work of the reporters by offering in advance any extended remarks upon questions which they are better prepared to discuss.

The foreign literature relating to the subject of fertilizer analysis, while somewhat abundant during the past year, presents, as far as my observation extends, little that is new, and the bulk of this literature is devoted to the consideration of only a few features of the subject.

The methods for the determination of phosphoric acid and potash have received, so it would appear, no little attention at the hands of foreign investigators, and yet a large proportion of the articles relating to the former substance treats of the analysis of Thomas slag and particularly of the citrate solubility of that material.

In the foreign literature, with reference to phosphoric acid determinations, we find quite a voluminous contribution by Meineke,¹ which treats of the results of a number of comparative tests of the leading methods in use and gives especial attention to a comparison of the processes of Wagner and Maercker as relates to the precipitation with

¹ Chem. Ztg., 20, 108.

magnesia mixture, while Foerster has presented quite a lengthy dissertation on the relation between the citrate solubility and soil solubility of phosphoric acid with especial reference to Thomas slag.

The Wagner citrate method, as it appears in the official methods of the German Manure Manufacturers' Association and as published in the *Chemiker Zeitung* (No. 63, 1895), appears to be meeting with quite extensive employment at present, and it is claimed that the analytical tests give results corresponding quite well with the actual soil availability.

The subject of the determination of the approximate availability of phosphoric acid in certain forms of nonacidulated phosphates has often been discussed by this association, and it has been frequently stated that our present citrate method fails to give altogether satisfactory results upon certain classes of raw phosphates.

A sample of Florida soft phosphate recently examined in the Alabama State Laboratory gave 0.78 per cent citrate soluble phosphoric acid by our association method, 0.31 per cent by Wagner's citrate method as applied to Thomas slag, and 0.84 per cent by Wagner's method as employed for precipitated phosphates and superphosphates.

These meager results would seem to indicate that the methods of Wagner have little advantage over our present method so far as phosphates of this character are concerned, and the details of the process for Thomas slag are more troublesome than the official method.

Of course, in matters involving modification of existing methods or the adoption of any supplementary methods, the association, as has been its wont, will proceed with great caution, and a thorough practical study of the subject would be necessary in advance of the adoption of any modifications of, or additions to, our present methods, even if the association should see fit to make any alterations.

The question of taking steps in the direction of securing greater harmony and a closer agreement between the official methods in use in this country and in the principal European countries received the favorable consideration of this association some three years since, and cooperation with foreign chemists has been obtained to a limited extent at least, during the period referred to, a number of samples each year having been placed in the hands of European analysts for comparative tests.

On the 27th of July the World's Congress of Applied Chemistry assembled in Paris in its second biennial session, and since one of the leading objects of this congress was to promote a closer agreement between, and a nearer approach to, uniformity in the analytical methods employed in the leading industries in the various countries represented, a statement as to the outcome of this convention would be of great interest.

The distinguished secretary of this association was in attendance upon the congress referred to, and has already presented in a recent

number of the Journal of the American Chemical Society an extremely interesting account of its proceedings and deliberations. I trust, however, that the association will be favored with a further statement as to the results of the congress and as to the prospects of harmonizing the methods of analysis at present employed in the principal countries of the world.

As stated in the above article of Dr. Wiley, and as reported in the résumé of the proceedings published in the Bulletin de L'Association des Chimistes de Sucrierie et de Distillerie, it was directed by vote of the congress that the official methods of Germany, France, and the United States be published in both the French and German languages, the work of editing and publication being placed in the hands of D. Sidersky, of Paris, the author of the well-known treatise on sugar chemistry.

The above journal also states that a commission, consisting of representatives of three different sections of the congress, was appointed to formulate or prepare methods for the determination of potash in the principal technical or commercial products containing this constituent.

The members of our association have no doubt been pleased to learn of the action taken with reference to the publication of our official methods in both French and German, as our methods have in the past been ignored to a great extent by chemists on the other side of the water. In a number of instances, methods adopted by our association or reported in our proceedings have appeared, after the lapse of some time, in foreign periodicals, possibly with some of the minor details modified slightly, if at all, and without any statement or acknowledgment as to the origin of the method thus reproduced. With our methods, however, published under the sanction of this congress of chemists in the two chief languages of Continental Europe, let us hope that transatlantic chemists will become better acquainted with our processes of analysis and will concede to them at least a share of the merit and recognition which is accorded them at home.

A recent report of a committee, appointed by the Belgian Association of Chemists to investigate the comparative merits of the leading methods for potash determination in use in the laboratories of that country, gives the results of tests of a number of processes, although the Lindo-Gladding method does not appear in the list of methods investigated. The results reported seem to indicate that there is quite a diversity in the methods employed in that country, and also that the same method in the hands of different analysts fails to give satisfactory and concordant results. The sample of fertilizer sent out for these cooperative tests had a known content of 5.40 per cent of water soluble potash, while the results reported by eight chemists vary from 4.69 to 6.51 per cent, a variation between the extremes of 1.82 per cent, amounting to about 33.5 per cent of the actual potash content, or about 39 per cent of the minimum result.

We should take courage from the examination of these figures, which

read somewhat like some of the association's past results for crude fiber or nitrogen free extract, and our organization is to be congratulated that, so far as our fertilizer work is concerned, the variations in results of our cooperative tests of samples have been reduced within limits much narrower than we had originally hoped to attain at so early a period of the association's existence.

A recent number of the *Bulletin de l'Association des Chimistes* makes mention of a new nitrogenous fertilizer calcium cyanate, which awaits employment at the hands of European agriculturists, but little information is given with regard to its probable economic value or utility.

The fertilizer control in some of our Southern States has experienced some little trouble during the past year or two on account of the sale or offering for sale of formulæ or receipes for the preparation of home-mixed manures designed to supplant or supersede the commercial article. In practically all such cases the formulæ are absolutely worthless, as they prescribe the use of such substances as ammonium nitrate, commercial ammonia, caustic potash, soda ash, bluestone, sulphur, free phosphoric acid, and various inert bodies or incompatibles which are mixed together and incorporated with some such material as ordinary earth or barnyard scrapings. In several of the States the sale of such fraudulent formulæ has been promptly exposed and the attention of the agricultural public called to the worthlessness of such recipes.

In accordance with a resolution of this association adopted at a former meeting, the work of the abstract committee has appeared in regular installments with each issue of the *Experiment Station Record*, and we are thus enabled to secure the benefits of foreign contributions to the literature of agricultural analysis, presented in a convenient and condensed form, and reaching us much more promptly than by the publication of such abstract matter in the same volume with our annual proceedings.

At the last annual meeting of this association the president in his opening address called attention to the importance of the study and investigation of foods for human consumption, and pursuant to a resolution adopted at that time a committee was appointed to consider what attitude it is desirable for this body to assume with reference to food adulterations and legislation relating thereto, and a report from this committee will no doubt be presented in due order.

Our methods for the analysis of dairy products, as they stand at present, are of course chiefly quantitative in their character, and although extremely comprehensive, do not include tests for the more common adulterants or preservatives encountered in such materials. It is true that methods covering these cases are within easy reach of every chemist who has access to reference works and periodical literature on the subject, but in cases of this character, which frequently come before the courts for adjudication, the findings of the chemist will be largely strengthened if it is known that the methods employed in

his tests have received the official sanction of an association of this character.

The investigations of the Division of Chemistry of the United States Department of Agriculture, continued through a number of years, have, however, supplied ample contributions to every department of the literature of foods and food adulterations, and a reference to its publications on this subject will supply the desired information in almost every individual case which may arise.

The Division of Nutrition Investigations has given much time and attention to the study of the composition of the chief food materials consumed in the various sections of this country, and has conducted a large number of dietary studies in the Northern, Western, and Southern States, the results of these investigations being embodied in a series of publications issued by the Department.

The matter of the analysis of foods and feed stuffs, as shown by the experience of this association, is one of the most difficult questions connected with the work of this organization, but with the progress already made in this line of work, and in view of the success already attained in other departments of association work, we have reason to hope for substantial improvement in the near future.

In the meantime, while the processes employed are doubtless the best within our reach at present, it is nevertheless to be conceded that, as regards some constituents, the methods provisionally in use constitute simply a *modus vivendi* until something better is secured.

In the consideration of the question of changes in our methods of analysis, the association will no doubt continue to pursue the policy it has adhered to in the past, and while we have not hesitated to alter or discard existing methods when it is shown by actual results and figures that another process is more desirable, our proceedings have, at the same time, been characterized by a spirit of conservatism which "makes us rather bear those ills we have than fly to others that we know not of."

MEMBERS AND VISITORS PRESENT.

The following members and visitors were present :

- Allen, W. M., Raleigh, N. C.
- Allen, E. W., Office of Experiment Stations, United States Department of Agriculture,
Washington, D. C.
- Armsby, H. P., Director Agricultural Experiment Station, State College, Pa.
- Bigelow, W. D., Division of Chemistry, United States Department of Agriculture,
Washington, D. C.
- Battle, H. B., North Carolina Experiment Station, Raleigh, N. C.
- Beal, W. H., Office of Experiment Stations, United States Department of Agriculture,
Washington, D. C.
- Buckley, S. S., Maryland Agricultural College, College Park, Md.
- Bomberger, F. B., Maryland Agricultural College, College Park, Md.
- Curtis, H. E., Lexington, Ky.
- Carr, Oma, Corry, Pa.
- Carpenter, F. B., Virginia-Carolina Chemical Company, Richmond, Va.

- Cutter, W. P., Librarian, United States Department of Agriculture, Washington, D. C.
- Caldwell, G. C., Cornell Experiment Station, Ithaca, N. Y.
- Crampton, C. A., Treasury Department, Washington, D. C.
- Clarke, F. W., Chief Chemist United States Geological Survey, Washington, D. C.
- Davidson, R. J., Virginia Agricultural Experiment Station, Blacksburg, Va.
- Dugan, J. A., Division of Chemistry, United States Department of Agriculture, Washington, D. C.
- Ewell, E. E., Division of Chemistry, United States Department of Agriculture, Washington, D. C.
- Frear, William, Agricultural Experiment Station, State College, Pa.
- Fraps, G. S., Johns Hopkins University, Baltimore, Md.
- Goss, Arthur, Mesilla Park, N. Mex.
- Gaines, R. H., State Chemist of Virginia, Richmond, Va.
- Herff, B. von, 99 Nassau street, New York, N. Y.
- Hills, J. L., Vermont Agricultural Experiment Station, Burlington, Vt.
- Hite, B. H., Morgantown, W. Va.
- Hird, J. D., Chemist District Board of Health, Washington, D. C.
- Huston, H. A., State Chemist of Indiana, Lafayette, Ind.
- Johnson, S. W., Yale University, New Haven, Conn.
- Kilgore, B. W., Raleigh, N. C.
- Langworthy, C. F., Office of Experiment Stations, United States Department of Agriculture, Washington, D. C.
- Laughlin, J. R., Maryland Agricultural College, College Park, Md.
- Lindsey, J. B., Hatch Experiment Station, Amherst, Mass.
- McDonnell, H. B., State Chemist, College Park, Md.
- Merrill, L. H., Orono, Me.
- Moore, C. C., Division of Chemistry, United States Department of Agriculture, Washington, D. C.
- Myers, J. A., Director Agricultural Experiment Station, Morgantown, W. Va.
- Patrick, G. E., Division of Chemistry, United States Department of Agriculture, Washington, D. C.
- Patterson, H. J., Agricultural Experiment Station, College Park, Md.
- Peacock, S., American Fertilizer Co., Philadelphia, Pa.
- Penny, C. L., Newark, Del.
- Perkins, W. R., Agricultural College, Mississippi.
- Quick, W. J., Columbia, Mo.
- Ross, B. B., Alabama Experiment Station, Auburn, Ala.
- Runyan, E. G., Division of Chemistry, United States Department of Agriculture, Washington, D. C.
- Street, J. P., Agricultural Experiment Station, New Brunswick, N. J.
- Schweinitz, E. A. de, Bureau of Animal Industry, United States Department of Agriculture, Washington, D. C.
- Scovell, M. A., Kentucky Experiment Station, Lexington, Ky.
- Shutt, F. T., Ottawa, Canada.
- Skinner, W., Maryland Agricultural College, College Park, Md.
- Teller, G. L., Agricultural Experiment Station, Fayetteville, Ark.
- Trescot, T. C., Division of Chemistry, United States Department of Agriculture, Washington, D. C.
- Van Slyke, L. L., Agricultural Experiment Station, Geneva, N. Y.
- Veitch, J. P., Maryland Agricultural College, College Park, Md.
- Wheeler, H. J., Chemist Rhode Island Experiment Station, Kingston, R. I.
- Wiley, Harvey W., Chief of Division of Chemistry, United States Department of Agriculture, Washington, D. C.
- Williams, C. B., Johns Hopkins University, Baltimore, Md.
- Winton, A. L., New Haven, Conn.
- Yocum, J. H., 30 Ferry street, New York, N. Y.

The PRESIDENT. Before proceeding to the regular order it will be necessary to appoint a committee on recommendations of reporters. The committee will consist of Messrs. Wheeler, Huston, Van Slyke, Gaines, and Kilgore.

Following the regular order, the reporter on nitrogen now has the floor.

Mr. STREET. I beg to present the following report:

REPORT ON NITROGEN.

By JOHN P. STREET.

The work of your reporter this year was largely influenced by suggestions made at our last convention. The Ulsch and zinc-iron methods for nitric nitrogen and the Fassbender method for total nitrogen were recommended for further trial. In addition to these lines of work it has seemed to me to be advisable to give some attention to the question of the availability of organic nitrogen, so that, even if no conclusive results were secured, this important question might at least be started toward a satisfactory solution. The report will therefore be considered under two heads:

I. New methods of determining nitrogen in commercial fertilizers.

II. A preliminary study of the availability of organic nitrogen.

Early in December a letter requesting cooperation was addressed to 60 chemists, representing all of the American experiment stations and several commercial firms. Twenty-four favorable replies were received to this letter, and of this number 16 have reported results either on all the work or in part. On July 30 a letter of instruction was sent to those who had signified a wish to take part in the work.

PREPARATION OF SAMPLES.

Three samples of fertilizers were prepared for distribution. The materials used in compounding these mixtures were sodium nitrate, potassium nitrate, tankage, cotton-seed meal, linseed meal, ammonium sulphate, acid phosphate, potassium chlorid, and sodium chlorid. These ingredients were dried for five hours at from 60° to 70° C., and were then allowed to stand exposed to the air for fifteen hours in order that they might become in equilibrium with the atmosphere. They were then ground to pass through a one twenty-fifth inch sieve and bottled. The salts were afterwards passed through a one-fiftieth inch sieve also. After grinding they were thoroughly mixed and samples for analysis taken, the results of which follow:

Nitrogen as—	Nitrates.	Ammonia.	Organic N.	Total N.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Sodium nitrate	15.89	15.89
Potassium nitrate	13.20	13.20
Tankage.....	0.20	12.06	12.26
Cotton-seed meal	7.69	7.69
Linseed meal	6.03	6.03
Ammonium sulphate	21.18	21.18
Acid phosphate.....13	.13

No traces of nitrogen were found in the potassium chlorid or sodium chlorid.

The ingredients of the different mixtures were then spread out on a heavy paper and thoroughly mixed, from one to two hours, by turning with a spatula and passing through a drug mill. They were then bottled and sealed, and the first, tenth, twentieth, and thirtieth bottles of each series were kept for the use of the reporter.

The mixtures were made up as follows:

Sample No. 1:

900 grams sodium nitrate, containing 143.01 grams nitrogen.
525 grams potassium nitrate, containing 69.30 grams nitrogen.
75 grams potassium chlorid.
Or 14.15 per cent nitric nitrogen.

Sample No. 2:

150 grams sodium nitrate, containing 23.83 grams nitrogen.
300 grams cotton-seed meal, containing 23.07 grams nitrogen.
300 grams linseed meal, containing 18.09 grams nitrogen.
600 grams acid phosphate, containing 0.78 grams nitrogen.
150 grams potassium chlorid.

Or 4.38 per cent total nitrogen, containing 1.59 per cent nitric nitrogen.

Sample No. 3:

225 grams sodium nitrate, containing 35.75 grams nitrogen.
75 grams ammonium sulphate, containing 15.89 grams nitrogen.
300 grams tankage, containing 36.78 grams nitrogen.
750 grams acid phosphate, containing 1.08 grams nitrogen.
150 grams potassium chlorid.

Or 5.96 per cent total nitrogen, containing 2.38 per cent as nitrates and 1.10 per cent as ammonia.

In preparing these mixtures, No. 1 was intended to represent a highly nitrated sample and Nos. 2 and 3 commercial fertilizers—one containing nitrates and the other both nitrates and ammonia, in connection with organic nitrogen.

As a check on the thoroughness and evenness of mixing, total nitrogen was made in portions from four different bottles of each sample with the following results:

The following table shows the percentage of total nitrogen in four determinations:

Sample.	A.	B.	C.	D.
No. 1.....	14.12	14.12	14.12	14.14
No. 2.....	4.39	4.40	4.41	4.39
No. 3.....	6.04	6.03	6.06	5.99

From these results it is evident that the samples left the reporter's hands thoroughly homogeneous and well mixed.

ULSCH-STREET METHOD FOR NITRATES.

The method recommended was slightly different from that published in the last report of the association, and is as follows:

Place 1 gram of material into a flat-bottomed 500 cc flask; add 2 to 3 grams of reduced iron, 25 cc of water, and 10 cc of sulphuric acid (sp. gr. 1.35). Shake well wash down the sides of the flask with a little water, and boil gently for five minutes. (Where the material is very rich in nitrates, as in nitrate of soda, the use of a dropping bulb is recommended.) Add about 100 cc of water, a little paraffin, and 3 to 5 grams of heavy oxid of magnesia: distill for thirty-five to forty minutes, nearly to dryness, and titrate as usual. The nitrogen obtained represents the nitrates, plus the ammonia, contained in the sample.

In the analysis of nitrate salts proceed as above, except that 25 cc of the nitrate solution are employed with 5 grams of reduced iron. After boiling add 75 cc of water and an excess of sodium hydrate, and proceed as above.

Cautions.—The magnesium oxid should be fresh and practically free from carbon dioxid. In the distillation bring to a boil very gradually, as at first there is a tendency to foam.

Percentage of nitrogen by Ulsch-Street method by distillation with NaOH and MgO.

Analyst.	No. 1.		No. 2.	No. 3.
	NaOH.	MgO.		NH ₃ de- ducted.
Wisconsin Station, F. W. Woll	13.85	1.59	12.67
Virginia Station:				
R. J. Davidson	13.78	1.62	2.43
A. T. Eskridge	13.77	1.63	2.44
W. B. Ellett	13.66	1.63	2.46
Maryland Agricultural College:				
F. P. Veitch	14.09	1.67	2.33
H. B. McDonnell	13.90	1.68	12.24
F. B. Bomberger	13.86	¹ 1.72	2.47
Illinois Station, C. G. Hopkins	14.02	13.98	¹ 1.70	2.31
Vermont Station, C. H. Jones		14.16	1.65	2.31
Pennsylvania Station:				
W. S. Sweetser	13.87	1.68	2.35
C. A. Browne	13.77	1.66	2.35
United States Department of Agriculture, T. C. Trescot	14.09	¹ 1.79	2.31
Lister's Agricultural Chemical Works, C. S. Cathcart	14.04	1.62	2.36
New Jersey Station, J. P. Street	14.15	1.62	2.38
Average			1.64	2.38
Theory	14.15	1.59	2.38

¹ Omitted from the averages.

COMMENTS OF ANALYSTS.

R. J. Davidson: Obtained 14.17, 14.17, and 14.22 in No. 1 by the following modification: To the 25 cc of solution taken, add 100 cc of water, 5 grams of reduced iron, and 15 cc of sulphuric acid (1.35); let stand until all action ceases, then heat for five minutes and add 100 cc of water, a little granulated zinc and paraffin, and 30 cc of sodium hydrate; distill almost to dryness. He experienced considerable difficulty from bumping when sodium hydrate was used.

Maryland Agricultural College: Preparatory to distillation the solutions were heated slowly for forty-five minutes, and then boiled for five minutes with the following results:

Percentage of nitrogen.

Analyst.	No. 1.	No. 2.	No. 3.
F. P. Veitch	14.22	1.67	2.56
H. B. McDonnell	14.17	1.72	2.49
F. B. Bomberger	14.11	1.70	2.37

Fourteen chemists reported results by the modified Ulsch method. The results secured in No. 1 are far from satisfactory. Of the 14 sets reported only six approach the calculated percentage. The results vary from 13.66 to 14.15 per cent, or 0.49 per cent. No trouble whatever was experienced in the New Jersey laboratory when the instructions sent out were strictly followed. If the analysts had followed the method strictly, especially the steps after the reduction was effected, it is difficult to understand why good results would not have been secured. Incomplete distillation is probably the chief cause of the low results. A brisk boiling of forty minutes, which is usually nearly to dryness, will cause all the ammonia to be distilled over. Some of the analysts reported difficulty with bumping when the solutions became low. Several hundred determinations by this method have been made in the New Jersey laboratory and we have never experienced this difficulty. The freshness of the magnesium oxid is a very important point. The instructions expressly stated that fresh oxid should be used for the liberation of the ammonia. We have had some trouble with

this method occasionally in the past, and have always found the cause to be impurity of the magnesia used. Exposure to the air causes it to carbonate, which not only decreases its effectiveness but renders the titration very difficult as well as inaccurate.

Several suggestions have been made also as to the preliminary treatment of the solution before and during the reduction period. The experience of our laboratory with the method is that the amount of acid used and the time of boiling have little to do with the final results. Whether 5, 10, or 15 cc of sulphuric acid are used, or whether the solution is boiled five minutes or one hour, matters little. The smaller amount of acid has been found, by actual trial, to be quite sufficient, and the larger quantity was recommended for the sole purpose of insuring an excess. As to the time of boiling or digesting, Ulsch, the originator of the method, has shown (*Zeit. Anal. Chem.*, 30, 175-182) that the conversion of the nitrates into ammonia is almost instantaneous, less than one minute being required for the operation. It will be seen, therefore, that an extended boiling of the solution theoretically should have little effect on the results. The concentration of the solution is also important. In all of our work with the Ulsch method, especially on nitrate of soda, we have endeavored to keep the solution as concentrated as possible. Our experience is that the greater the concentration is, the less difficulty there is in securing a complete distillation of the ammonia.

It is to be regretted that the results on this sample are no better. The method has been before the association for nearly five years and has been used for that period by the New Jersey Station, where it has given perfect satisfaction. The results this year would seem to indicate that the analysts have not followed the method exactly as given in all its details, probably omitting some apparently unimportant step, on which however may depend the method's success. It is also instructive to notice that six of the analysts, who reported low results on this sample by the Ulsch method, had quite as much difficulty in securing satisfactory results by the Kjeldahl and Gunning methods, both of which are accepted methods of this association. Their results by these methods varied from 0.28 to 0.71 per cent less than the theoretical composition. One analyst reported a result 0.50 per cent below theory, and while requesting him to repeat his work I asked for a portion of his sample that I might test it also. My three results were 14.13, 14.10, and 14.10 against 14.15 theory. This would seem to offer additional testimony as to the uniformity of the sample sent out.

The results obtained by this method in samples 2 and 3 are all that could be desired. In sample 2 only 3 of the 14 chemists vary from theory by more than 0.10 per cent, while the greatest variation is 0.20 per cent; in sample 3, only 2 of the 14 vary more than 0.09 per cent, one being 0.29 per cent higher than the other 0.14 per cent lower than theory. These results show that in mixed fertilizers at least the modified Ulsch method is an excellent working method, rapid and accurate.

ZINC-IRON METHOD.

The method recommended for trial was that given in Bulletin 46 of the United States Department of Agriculture, page 22.

Percentage of nitrogen.

Analyst.	No. 1.	No. 2.	No. 3.
Virginia Station:			
R. J. Davidson.....	13.80	1.68	2.30
A. T. Eskridge.....	13.66	1.63	2.40
W. B. Ellett.....	13.83	1.62	2.35
Illinois Station, C. G. Hopkins.....	14.03	1.66	2.38
Vermont Station, C. H. Jones.....	14.16	1.67	2.30
United States Department of Agriculture, T. C. Trescott.....	13.53	2.30
Averages.....	13.84	1.66	2.34
Theory.....	14.15	1.59	2.38

Only six chemists reported results by this method. The work on sample 1 is quite as unsatisfactory, if not more so, than that secured by the Ulsch method on the same sample, only two chemists approaching theory, while there is a variation from 13.53 to 14.16 per cent, or 0.63 per cent. In samples 2 and 3 the results were much better, the variations being slight, and all quite near the average. The reporter has repeatedly tried this method during the past three years and has never been able to secure satisfactory results with it. In samples containing much organic matter there is a very decided tendency to foam, and during the distillation it is almost impossible to prevent the mechanical carrying over of alkali. The results on samples 2 and 3, while good, are by no means conclusive. The source of their organic nitrogen was cotton-seed meal, linseed meal, and tankage, all of which are quite refractory materials. On samples containing dried blood and other materials whose nitrogen is easily decomposed, it would seem that some of the organic nitrogen would pass over in the distillation, thus giving high results. It was this tendency which prompted the reporter to modify the Ulsch method, so that magnesium oxid was used in place of sodium hydrate. The reporter regrets that through inadvertence, in the preparation of his samples, he overlooked this method, so that it will be impossible to deduce from the results secured any definite conclusions.

FASSBENDER METHOD.

The method recommended by the reporter was as follows:

Place 1 gram of material into a Kjeldahl flask, cover with 20 cc of water, shake and allow to stand for ten minutes; then add 2 grams of powdered iron (reduced iron answers very well) and 10 cc of sulphuric acid (sp. gr. 1.35). Warm gently on furnace until evolution of hydrogen ceases. Add 0.7 gram of mercuric oxid and 20 cc of strong sulphuric acid, and proceed as in the Kjeldahl method.

Percentage of nitrogen.

Analyst.	No. 2.	No. 3.
Lister's Agricultural Chemical Works, C. S. Cathcart.....	4.27	5.96
Virginia Station:		
R. J. Davidson	4.37	¹ 5.65
A. T. Eskridge.....		5.87
W. B. Ellett.....	4.53	¹ 5.65
Maryland Agricultural College:		
F. P. Veitch.....	4.39	5.99
H. B. McDonnell.....	4.34	5.93
F. B. Bomberger.....	4.37	5.91
Illinois Station, C. G. Hopkins.....	4.44	6.18
North Carolina Station, W. M. Allen.....	4.31	6.02
Vermont Station, C. H. Jones.....	¹ 3.77	¹ 4.41
Pennsylvania Station:		
W. S. Sweetser.....	4.21	5.75
C. A. Browne.....	4.21	5.73
New Jersey Station, J. P. Street.....	4.40	6.01
Connecticut Station, A. L. Winton.....	¹ 4.41	¹ 5.95
Averages.....	4.35	5.94
Theory.....	4.38	5.96

¹ Not included in the averages.

Thirteen chemists reported results by this method. The results are fairly satisfactory, but not as close as they might be. In sample 2 the results of four analysts vary more than 0.10 per cent from the average, while only one varies more than 0.18 per cent. When it is remembered that a very large proportion of the nitrogen of this sample is derived from cotton-seed and linseed meal, this variation is not so surprising.

In sample 3, omitting the three results varying more than 0.25 per cent from the average, we find seven chemists reporting results less than 0.09 per cent from theory, while three vary from 0.21 to 0.24 per cent. The work by the Fassbender method on these samples is, on the whole, all that could be expected from the first trials of an unfamiliar method. The reporter is satisfied that with it, as with the Ulsch method, when failure to obtain good results occurs, it is usually due to lack of practice in the manipulation of the method.

At the last meeting of the association the nitrogen reporter called attention to a number of comparative results obtained by this method by Mr. L. H. Merrill, of the Maine Station, and by myself. These tests were secured in our regular fertilizer work and, as the results show, were very satisfactory. In 32 samples Mr. Merrill secured an average of 2.39 per cent by the Gunning and 2.37 per cent by the Fassbender method. In 107 samples I obtained an average of 2.96 per cent by the modified Kjeldahl and 2.99 per cent by the Fassbender method. In addition to these results I have made during the past season some additional comparative tests which follow:

Per cent of nitrogen obtained by different methods.

Official.	Fassbender.	Official.	Fassbender.	Official.	Fassbender.	Official.	Fassbender.	Official.	Fassbender.
2.51	2.44	3.15	3.10	1.06	0.98	1.66	1.67	3.51	3.53
2.58	2.51	2.77	2.65	1.93	1.83	2.04	1.97	3.07	3.85
2.50	2.58	3.29	3.28	1.74	1.65	1.36	1.28	1.82	1.74
3.78	3.69	2.04	2.05	2.88	2.78	3.97	4.08	2.25	2.18
3.92	3.92	1.55	1.49	1.69	1.60	2.66	2.65	2.97	2.95
4.33	4.30	3.11	3.12	2.96	2.86	2.42	2.49	4.52	4.53
3.11	3.06	2.45	2.49	2.04	2.06	3.35	3.32	1.57	1.53
3.15	3.07	3.30	3.33	3.60	3.51	2.70	2.63	1.99	2.00
3.34	3.44	2.59	2.49	1.75	1.69	1.83	1.81	1.47	1.37
3.05	2.95	2.91	2.84	3.53	3.43	2.01	1.97	2.47	2.49
3.84	3.82	1.66	1.60	3.18	3.06	3.38	3.41	2.66	2.55

Averages: Official, 2.69; Fassbender, 2.65.

Fifty-five comparative tests were made, by which an average of 2.69 per cent was obtained by the official method and 2.65 per cent by the Fassbender. The samples tested represented regular commercial fertilizers, and the analyses were all made in the rush of routine work.

MISCELLANEOUS RESULTS.

Some of the analysts reported results which did not properly come under the discussion in the preceding sections, and which I tabulate below:

Per cent of nitrogen obtained by different methods.

Analyst.	No. 1.			No. 2.		No. 3.	
	Kjel-dahl.	Gunning.	Fassbender.	Kjel-dahl.	Gunning.	Kjel-dahl.	Gunning.
Lister's Agricultural Chemical Works.							
C. S. Cathcart.....	14.02			4.33		5.96	
Wisconsin Station, F. W. Woll.....				4.44		5.83	
Virginia Station.							
R. J. Davidson.....				4.36	4.33	6.02	6.08
A. T. Eskridge.....					4.43	5.97	5.87
W. B. Ellett.....					4.42		5.90
Maryland Agricultural College:							
F. P. Veitch.....		13.44			4.28		5.94
H. B. McDonnell.....					4.14		5.78
F. B. Bomberger.....		13.74	14.22		4.16		5.73
Illinois Station, C. G. Hopkins.....	14.06		13.98	4.45		6.12	
North Carolina Station, W. M. Allen.....	14.07			4.33		6.06	

Per cent of nitrogen obtained by different methods—Continued.

Analyst.	No. 1.			No. 2.		No. 3.	
	Kjel-dahl.	Gunning.	Fass-bender.	Kjel-dahl.	Gunning.	Kjel-dahl.	Gunning.
Rhode Island Station, B. L. Hartwell.....	-----	-----	-----	4.35	-----	6.01	-----
Vermont Station, C. H. Jones.....	-----	-----	-----	4.29	-----	5.85	-----
Pennsylvania Station:							
W. S. Sweetser.....	14.05	-----	-----	4.33	-----	5.72	-----
C. A. Browne.....	¹ 13.87	-----	-----	4.31	-----	5.77	-----
United States Department of Agriculture,							
T. C. Trescot.....	-----	-----	-----	-----	4.40	5.97	5.98
New Jersey Station, J. P. Street.....	14.14	-----	-----	4.39	4.36	6.03	6.04
Averages	14.07	13.59	14.12	4.36	4.32	5.94	5.92
Theory.....	14.15	-----	-----	4.38	-----	5.96	-----
Connecticut Station, A. L. Winton.....	-----	-----	-----	4.40	4.37	5.94	5.96

¹ Omitted from the average.

The results in No. 1 by the modified Kjeldahl method are very satisfactory, especially when it is remembered that all the nitrogen is in the nitric form. Five of the six results reported vary less than 0.10 per cent from theory.

The results by the Gunning and Fassbender methods are too few to warrant any conclusions, although the Gunning results are somewhat disappointing in light of the fact that the method has already been made official. In Nos. 2 and 3 the results by both the modified Kjeldahl and Gunning methods are very good, with two or three exceptions. In No. 2 the result by the Kjeldahl method varies more than 0.10 per cent from theory, while only two by the Gunning show such a variation. In No. 3 five of the twelve results reported vary more than 0.10 per cent, but only two more than 0.20 per cent from theory. Two of the analysts experienced difficulty with the Gunning method in all three samples, as they did with the Ulsch method also.

MAGNESIUM-OXID METHOD.

Percentage of nitrogen.

Analyst.	No. 3.
Lister's Agricultural Chemical Works, C. S. Cathcart.....	1.09
Wisconsin Station, F. W. Woll.....	1.00
Virginia Station:	
R. J. Davidson.....	1.11
A. T. Eskridge.....	1.00
W. B. Ellett.....	1.07
Maryland Agricultural College:	
F. P. Veitch.....	1.07
H. B. McDonnell.....	1.07
F. B. Bomberger.....	1.10
Illinois Station, C. G. Hopkins.....	1.05
Rhode Island Station, B. L. Hartwell.....	1.08
Vermont Station, C. H. Jones.....	1.01
Pennsylvania Station:	
W. S. Sweetser.....	1.05
C. A. Browne.....	1.05
United States Department of Agriculture, T. C. Trescot.....	1.10
New Jersey Station, J. P. Street.....	1.09
Average.....	1.06
Theory.....	1.10

Fifteen analysts determined the nitrogen existing in the form of ammonia in No. 3 by the magnesium-oxid method. No result varies more than 0.10 per cent from theory and only three more than 0.05 per cent.

QUALITATIVE DETERMINATION OF NITRIC NITROGEN.

It is of considerable importance to the farmer that he should know in what form the nitrogen which he buys exists. It is also of importance to this association that the results of its analyses should convey to the farmer just as much definite information as possible. The difference in cost to the manufacturer of the different forms of nitrogenous matter furnishes another argument for this differentiation of the forms of nitrogen. The average wholesale prices per pound of raw materials during 1895 were 11, 13.5 and 11.2 cents per pound for nitric, ammoniacal, and organic nitrogen, respectively. Of the 331 complete fertilizers analyzed in New Jersey this year 187, or 56 per cent, contained nitrogen in the nitric form and 104, or 31 per cent, contained nitrogen derived from ammonia salts. As long as these variations in composition and cost exist it would seem only just, both to the manufacturer and the consumer, that the proper division should be made. While this is done in most of the stations, the practice is by no means universal. In the light of these facts the reporter would recommend the association to provide that in all fertilizer work these three forms of nitrogen should be determined.

Inasmuch as most of the stations do follow this practice, it has seemed to the reporter that the official methods would be more complete were a qualitative method for nitric nitrogen inserted in our report. The following method, based on well-known principles, is therefore recommended:

Five grams of the fertilizer are mixed with 25 cc of hot water and filtered. To a portion of this solution add two volumes of concentrated sulphuric acid, free from nitric acid and oxids of nitrogen, and allow the mixture to cool. Add cautiously a few drops of a concentrated solution of ferrous sulphate, so that the fluids do not mix. If nitrates are present, the junction shows at first a purple, afterwards a brown color, or if present only in a very minute quantity, a reddish color. Another portion of the solution may be taken, and 1 cc of a dilute solution of nitrate of soda (3 grams to 300 cc) be added and tested as before to determine whether sufficient sulphuric acid has been added in the first test.

THE AVAILABILITY OF ORGANIC NITROGEN.

The increase of the fertilizer trade and the consequent seizure of any waste materials available for use in that trade have offered in some cases a temptation to certain manufacturers to make use of raw products, which, while they would show little effect in field practice, would nevertheless obtain a good analysis in the chemical laboratory. This tendency, although it may not be alarming at present, if developed as it may be, and as there are many reasons to believe that it will be, finds us without proper methods for its detection and correction. We have the means to determine how much of the total nitrogen exists as nitrates or ammonia, but the origin of the organic nitrogen is not revealed to us in our ordinary methods of analysis. Our present methods do not attempt to indicate whether the source is blood, tankage, cotton-seed meal, flesh, fish, or bone, all of which are valuable fertilizing materials, or whether it is garbage, tankage, leather, or wool waste, which are comparatively worthless.

This question is not a new one to American chemists, but up to the present time no method has been devised which gives universal satisfaction. While it is admitted that the only true test is one in which the fertilizers are tried on actual crops, and nature allowed to be the interpreter, still such a method is so time consuming as to be impracticable in most of our laboratories. While it is impossible to imitate exactly the action of the soil on a small amount of fertilizing material, the relative solubility of the materials under examination can be determined in the laboratory and compared with their relative crop-producing power. Then, as Dr. Jenkins says,

"if these two methods of examination give concordant results, the first named may be adopted as a test of the availability of nitrogen in mixed goods, which from the nature of the case could not be tested by the method of field or pot experiment."

Stutzer and Klinkenberg in 1882 suggested a method where the digestion was effected by a solution of pepsin. Drs. Shepard and Chazal, of South Carolina, applied the method to some peculiarly American ammoniates, and Dr. Jenkins, of Connecticut, has carried the work still further by also testing the method of putrefaction fermentation proposed by Morgen.

The conclusions from the experiments of these investigators are given in the report of the Connecticut Station for 1893, which I quote:

1. The nitrogen of dried blood, cotton seed, castor pomace, and maize refuse was in every case soluble in pepsin-hydrochloric acid, by twenty-four hours' digestion, to the extent of 75 per cent or more.

2. The nitrogen of fish, dried animal matter, and of bone was in every case soluble to the extent of over 52 per cent.

3. The nitrogen of leather, steamed or extracted by benzine, was in no case soluble to the extent of over 36 per cent. That of horn shavings, ground horn and hoof, cave guano, felt waste, and wool waste was considerably less soluble than the nitrogen of leather.

4. This method divides the organic nitrogenous matters used in fertilizers into two classes. In one, more than one-half of the nitrogen is soluble; in the other, scarcely more than one-third is soluble. To the first class belong all the materials whose nitrogen is known to be readily available in the usual sense. Of the second class the most soluble are leathers variously manipulated, which are comparatively worthless as ingredients of commercial fertilizers. To some extent this method is therefore a measure of the agricultural value of nitrogen. How far it is a measure must be determined by vegetation experiments under accurately controlled conditions, in which nitrogen is supplied in the same materials which have been tested by digestion experiments.

In the meantime the method has decided value, because in many cases it will distinguish in mixed fertilizers between such forms of nitrogen as the general sense of practical farmers accepts as available and such as the same tribunal condemns as inert.

After a carefully conducted series of vegetation tests in a large number of nitrogenous materials and tests by the pepsin-hydrochloric acid method in the same, Dr. Jenkins has come to the conclusion that, in general, the solubility of the nitrogen of these samples in pepsin solution was a fairly good measure of the relative availability of the nitrogen to the plant under the conditions of the vegetation experiment. In horn and hoof and dissolved leather, however, the amount of nitrogen dissolved by the pepsin was no indication of its availability.

PEPSIN TESTS IN COMPLETE FERTILIZERS.

In 1893 the Connecticut Station tested every mixed fertilizer analyzed by the station that year with pepsin-hydrochloric acid to determine the solubility of its organic nitrogen in that reagent. One hundred and twenty-five brands were examined with the following result:

In 3 brands over 90 per cent of the nitrogen were soluble in pepsin.

In 22 brands between 80 and 90 per cent of the nitrogen were soluble in pepsin.

In 62 brands between 70 and 80 per cent of the nitrogen were soluble in pepsin.

In 18 brands between 60 and 70 per cent of the nitrogen were soluble in pepsin.

In 12 brands between 50 and 60 per cent of the nitrogen were soluble in pepsin.

In 6 brands between 40 and 50 per cent of the nitrogen were soluble in pepsin.

In 2 brands under 40 per cent of the nitrogen were soluble in pepsin.

A fertilizer showing 60 per cent of its organic nitrogen soluble in pepsin may be considered as above suspicion, and one falling as low as 50 per cent must not necessarily be condemned, while one below 50 per cent must be regarded as suspicious. From the above tabulation it will be seen that in 1893 eight samples, or 6 per cent, of the mixed fertilizers sold in Connecticut fell in the suspicious class.

In order to determine whether the same condition existed in New Jersey, I tested in the same manner 49 brands sold in our State in 1895. I was limited in my tests

by lack of time, and while the number of fertilizers tested represents only a small proportion—18 per cent of the total number analyzed during the year—43 manufacturers are represented, and it is believed that the results secured are indicative of the average composition of New Jersey samples.

One gram of the fertilizer was washed on a filter with 250 cc of cold water to remove all soluble salts, and the residue was treated with the pepsin solution as hereinafter described. The results follow:

In 3 brands between 80 and 90 per cent of the nitrogen were soluble in pepsin.
 In 13 brands between 70 and 80 per cent of the nitrogen were soluble in pepsin.
 In 16 brands between 60 and 70 per cent of the nitrogen were soluble in pepsin.
 In 11 brands between 50 and 60 per cent of the nitrogen were soluble in pepsin.
 In 4 brands between 40 and 50 per cent of the nitrogen were soluble in pepsin.
 In 2 brands under 40 per cent of the nitrogen were soluble in pepsin.

It will be seen from the above that six, or 12 per cent, of the fertilizers examined have such a low availability that they must be regarded with suspicion. The results in Connecticut and New Jersey therefore agree in indicating that there are suspicious mixed fertilizers on the market. Their number, however, is not great, and those in New Jersey, at least, represent, with one exception, the product of small manufacturers whose product does not exceed 50 tons per annum.

Nevertheless the existence of these few suspicious fertilizers led your reporter to prepare some special mixtures and test them still further with pepsin and such other reagents as had been brought to his attention. Five samples were therefore prepared as follows:

Sample 4.—Dried blood, 90 grams; acid phosphate, 290 grams; muriate of potash, 70 grams, containing 2.92 per cent nitrogen, 8.26 per cent available phosphoric acid, and 7.86 per cent potash.

Sample 5.—Tankage, 108 grams; acid phosphate, 272 grams; muriate of potash, 70 grams, containing 3.04 per cent nitrogen, 7.74 per cent available phosphoric acid, and 7.86 per cent potash.

Sample 6.—Hoof meal, 85 grams; acid phosphate, 295 grams; muriate of potash, 70 grams, containing 2.58 per cent nitrogen, 8.40 per cent available phosphoric acid, and 7.86 per cent potash.

Sample 7.—Raw leather, 180 grams; acid phosphate, 200 grams; muriate of potash, 70 grams, containing 2.57 per cent nitrogen, 5.92 per cent available phosphoric acid, and 7.86 per cent potash.

Sample 8.—A suspicious-looking complete fertilizer sold in the State by one of the leading manufacturers. It contained small pieces of material strongly resembling leather in structure and appearance. It contained 2.03 per cent nitrogen, 8.21 per cent available phosphoric acid, and 6.04 per cent potash.

In the preparation of these samples it was intended to keep the relative amounts of the different fertilizing elements in about the same proportion. The nitrogen and potash were practically the same in samples 4, 5, 6, and 7, while the phosphoric acid varied from 5.92 to 8.40 per cent.

Before the samples were sent out Dr. Jenkins very kindly consented to conduct a series of vegetation tests with oats, using the samples I had prepared. These tests were made in pots such as are described in the Connecticut Report for 1893, page 231, and in the same kind of artificial soil as there described. To the artificial soil were added 5 grams of carbonate of lime, but it was believed that each of the fertilizers supplied enough of phosphoric acid and potash for the growth of the crop.

In the following table are given the amounts of nitrogen supplied, the weights of the separate crops, the nitrogen contained in them, and the percentage availability of the organic nitrogen. The table shows only what the crops have taken up and does not include the roots. There remains, of course, a certain amount of nitrogen in the soil in the form of roots, but it has been assumed, from the light of other experiments, that this residue is not large.

Vegetation experiments on oats.

No. of pot.	Nitrogen supplied.		Crop harvested (exclusive of roots).				Nitrogen increase.	Per cent of the fer-tilizer-nitrogen assimu-lated.	Average availabil-ity of ni-trogen.	
	In form of—	Quantity of nitro-gen sup-plied.	Weight of air-dry crop.	Weight of water-free crop.	Per cent of ni-trogen in air-dry crop.	Total ni-trogen of crop.				
		Grams.	Grams.	Grams.		Grams.	Grams.			
251	No. 4, dried blood	0.5	70.8	62.16	0.40	0.283	0.224	44.8	46.5	
252		.5	83.7	72.98	.36	.301	.242	48.4		
253		1.0	118.4	101.99	.43	.509	.450	45.0		
254		1.0	111.6	95.34	.48	.536	.477	47.7		
255	No. 5, tankage..	.5	78.5	69.10	.35	.275	.216	43.2	44.8	
256		.5	93.3	80.91	.33	.308	.249	49.8		
257		1.0	125.7	108.45	.43	.541	.482	48.2		
258		1.0	101.7	88.94	.43	.437	.378	37.8		
259	No. 6, hoof meal.	2.0	141.0	125.36	.77	1.086	1.027	51.3	43.4	
260		.5	76.1	66.22	.35	.266	.207	41.4		
261		.5	75.0	66.03	.34	.255	.196	39.2		
262		1.0	117.8	102.18	.44	.518	.459	45.9		
263	No. 7, raw leather.	1.0	112.7	96.74	.47	.530	.471	47.1	3.4	
264		2.0	132.1	117.93	.66	.872	.813	40.6		
265		.5	20.8	18.07	.36	.074	.015	3.0		
266		.5	21.7	18.91	.34	.074	.015	3.0		
267	No. 8, suspicious fertilizer.	1.0	24.3	21.51	.41	.100	.041	4.1	29.8	
268		1.0	26.0	22.54	.36	.094	.035	3.5		
269		.5	58.2	51.59	.36	.210	.151	30.2		
270		.5	60.1	53.19	.33	.198	.139	27.8		
271	No nitrogen added.	1.0	95.3	82.28	.36	.343	.284	28.4	29.8	
272		1.0	96.4	82.90	.40	.386	.327	32.7		
273		16.6	14.51	.32	.053	Average, .059		
274		18.4	16.00	.34	.063				
275		17.2	15.00	.35	.060				
276		16.2	14.21	.38	.062				

The average availability of the nitrogen shown in the table indicates that the nitrogen of the blood, tankage, and ground hoof in this experiment had practically the same availability, varying from 43.4 per cent in the case of horn to 46.5 per cent in the case of blood. The nitrogen of the raw leather had an availability of but 3.4 per cent, while the suspicious fertilizer confirms our suspicions with the low availability of 29.8 per cent.

From the following table, which shows the average results obtained by Dr. Jenkins on maize and oats with a number of nitrogenous materials, it will be seen that the new results agree very closely with the averages indicated.

	Per cent of available N reckoned on total N. Average of two years.	Same for new experiment.	Average per cent of three experiments.	Per cent of available N reckoned on available N of NaNO_3 .
Nitrate of soda	68.4	68.4	100
Castor pomace	50.6	50.6	74
Cotton-seed meal	49.7	49.7	72
Linseed meal	47.1	47.1	69
Dried blood	46.5	46.5	46.5	68
Dried fish	45.0	45.0	66
Dissolved leather	44.7	44.7	64
Ground hoof	42.5	43.4	42.8	63
Tankage	40.5	44.8	42.0	61
Steamed leather	6.5	6.5	9
Roasted leather	6.5	6.5	9
Raw leather	1.5	3.4	2.0	3

In order to find out what relation the availability secured by chemical methods had to that secured by these vegetation tests, samples of the five mixtures prepared for this work were sent to five chemists, who had had some experience with the methods proposed.

Two methods were proposed for trial—the pepsin-hydrochloric acid method and the Hayes potassium permanganate method.

PEPSIN-HYDROCHLORIC ACID METHOD.

The method of pepsin digestion recommended was substantially the same as described in the Connecticut Station Report for 1893, page 219, and is as follows:

Bring 1 gram of substance into a 150 cc flask, and add 100 cc of pepsin-hydrochloric acid solution. Place the flask, loosely corked, in a water-bath having a constant temperature of 40° C. Digest at this temperature for twenty-four hours, adding 2 cc of a 10 per cent hydrochloric acid solution at the end of the second, fifth, eighth, and eleventh hours. Shake the solution well on each addition of acid. At the end of the digestion transfer the contents of the flask to a filter, wash with 150 to 200 cc of cold water and determine nitrogen by the Kjeldahl method in the residue.

Pepsin-hydrochloric acid solution.—Mix 5 grams of pulverized pepsin (guaranteed to dissolve 2,000 times its weight of coagulated white of egg) in 1,000 cc of hydrochloric acid diluted to a strength of two-tenths per cent.

These analysts have reported results by this method as follows:

Analysts.	4.			5.			6.			7.			8.		
	Per cent total N.	Per cent soluble.	Per cent available.	Per cent total N.	Per cent soluble.	Per cent available.	Per cent total N.	Per cent soluble.	Per cent available.	Per cent total N.	Per cent soluble.	Per cent available.	Per cent total N.	Per cent soluble.	Per cent available.
R. J. Davidson.....	2.94	2.60	88.4	2.87	2.12	73.9	2.58	*1.07	*41.5	2.46	0.34	13.8	2.01	1.06	50.5
E. H. Jenkins	2.88	2.73	94.8	2.98	2.34	78.5	2.63	1.51	56.3	2.57	.45	17.5	2.15	1.25	58.1
J. P. Street	2.92	2.75	94.2	3.04	2.35	77.3	2.58	1.46	56.6	2.57	.48	18.7	2.03	1.05	51.7
Average	2.91	2.69	92.5	2.96	2.27	76.6	2.61	1.49	56.5	2.53	.42	16.7	2.06	1.12	53.4

* Omitted from average.

When it is remembered that such small quantities of the materials are used, and that a difference of a few hundredths will make a difference of several per cent in the per cent of availability, the above results are extremely satisfactory, and show that the method is capable of giving concordant results in the hands of different analysts. The value of this method, however, must be determined largely by the correspondence of its results with those secured by the vegetation tests. Reducing the data to a comparable basis we have the following:

Percentage of nitrogen.

Sample.	By vegetation test.	By pepsin digestion.
No. 4, blood.....	46.5	46.5
No. 5, tankage.....	44.8	38.5
No. 6, hoof.....	43.4	28.4
No. 7, raw leather	3.4	8.4
No. 8, fertilizer	29.8	27.8

The agreement between the two methods is quite satisfactory, except in the case of hoof meal. These tests confirm what Dr. Jenkins has already shown, namely, that the amount of nitrogen dissolved by pepsin is no measure of the availability of the nitrogen of hoof meal.

HAYES PERMANGANATE OF POTASH METHOD.

This method was suggested in a paper read at the last meeting of this association, and consists of treating 1 gram of material in a flask with 100 cc of potassium permanganate solution containing 16 grams KMnO_4 and 200 grams KOH per liter. Digest at a low temperature for one hour, then increase the temperature and distill for one hour, and titrate as usual.

The reporter was not able, from lack of time, to give the method an extended test before sending out samples, and since then has been able to do little additional work for the same reason. However, the few results obtained by Mr. Davidson and myself seem to indicate that there are possibilities in the method and that it is worthy of future study.

The following tabulation shows how the results secured agree with those by the vegetation tests:

Percentage of nitrogen.

Sample.	Davidson.	Alkaline, Street.	Solution, average.	Acid solution, Davidson.	Vegetation tests.
No. 4	46.5	46.5	46.5	46.5	46.5
No. 5	50.4	42.2	46.3	47.6	44.8
No. 6	58.2	51.4	54.8	41.8	43.4
No. 7	23.7	24.7	24.2	13.6	3.4
No. 8	35.8	32.3	34.0	33.6	29.8

The average results compared with the vegetation tests agree fairly, showing the method to give rather high results on hoof meal and raw leather, but approaching the truth nearer than the pepsin method. Mr. Davidson also made some tests where an acid solution of permanganate was used. His results are included in the above table and show quite a remarkable agreement with the vegetation tests. The method promises well and is worthy of further study in connection with other nitrogenous materials.

RECOMMENDATIONS

1. That the Ulsch-Street method for the determination of nitric nitrogen in mixed fertilizers be made an official method.
2. That the Fassbender method for the determination of total nitrogen be made an official method.
3. That the nitrogen reporter for 1897 be requested to investigate further the permanganate of potash method for determining the availability of organic nitrogen; also, the Ulsch method for determining nitrates in nitrate salts.
4. That it is the sense of this association that in reporting fertilizer analyses its members should indicate whether the nitrogen present exists as nitrates, ammonia, or as organic matter.
5. That a method for the qualitative determination of nitric nitrogen in fertilizers be included under the nitrogen methods.

AMENDED AND ADOPTED REPORT OF THE COMMITTEE ON RECOMMENDATIONS IN RELATION TO NITROGEN.

It is recommended—

- (1) That the Ulsch-Street method be referred to the reporters for the next year, to be further tested with special reference to nitrates.
- (2) That the method heretofore referred to as the Fassbender method be referred to the reporters for the next year for further trial, with request that they look up the question of whether the method should legitimately be called the Fassbender method or should be otherwise designated.

(3) That the Hayes permanganate method of determining the availability of organic nitrogen be referred to next year's reporters for further trial.

(4) That the following resolution be adopted: *Resolved*, That it is the sense of this association that the forms of the nitrogen in commercial fertilizers should be reported where it is possible to do so.

(5) That the qualitative method for the detection of nitrates by use of ferrous sulphate be adopted.

(6) That the comparison of the Tiemann-Schulze and Ulsch-Street methods for nitrates be referred to the reporters on nitrogen for 1897.

(7) That on page 21 under (h), third line from the top of the paragraph, be substituted for the words "a large excess of magnesia" the following: "5 grams or more of magnesium oxid free from CO_2 ."

H. J. WHEELER,

For Committee on Reporters' Recommendations.

The PRESIDENT. It is customary to appoint a committee to wait upon the Secretary and Assistant Secretary of Agriculture and invite them to attend our meetings. In accordance with the custom I will appoint Messrs. Frear and Lindsey on that committee.

Are there any papers on the subject of the determination of nitrogen?

Mr. SWEETSER presented the following:

COMPARISON OF NITROGEN DETERMINATION BY THE KJELDAHL METHOD, DISTILLING WITH AND WITHOUT POTASSIUM SULPHID.

By W. S. SWEETSER.

The average of 53 determinations of nitrogen in a miscellaneous lot of fertilizers by the official Kjeldahl method was 1.59 per cent, and by distilling without potassium sulphid, 1.61 per cent. With the official method as the basis, the omission of the potassium sulphid solution gave higher results in 29, lower in 10, and the same in 14 cases out of the 53. In 36 cases the variation was less than 0.05 of 1 per cent.

The distillations were made in copper flasks without the addition of zinc. The samples consisted of mixed fertilizers and a few ground bones. Some contained nitrates in abundance, and these were done by the Scovell method.

The results in all cases seem to indicate that the addition of potassium sulphid is unnecessary, especially when copper distilling flasks are used.

There being no additional papers on the subject of nitrogen determination, the President asked for the reading of the recommendations of the reporter. They were read as given above.

Mr. STREET. I desire to call special attention to the recommendation concerning the various forms of nitrogen, and to the importance of the analyst determining in what particular form the nitrogen exists.

The PRESIDENT. It is now in order to discuss the report and recommendations. The latter will be referred to the committee on recommendations, according to the regular order.

Mr. HUSTON. The reporter comments on the fact that the Ulsch-Street method gives poorer results in sample No. 1, and better in Nos. 2 and 3. This, however, is only apparently so, for when judged by the percentage of error the results in No. 1 are better than Nos. 2 and 3. The percentage of the error and the actual magnitude of the error should always be considered.

Mr. WINTON. I like the Ulsch-Street method for mixed fertilizers. I would also be glad to see the Schulze-Tiemann process tried, and would like the reporter next year to consider that method. It has been used very extensively, both here and in Europe, and it does not seem to me we should let so good a method fall into disuse.

Mr. WHEELER. I have used the Schulze-Tiemann method, and have an impression that it was once adopted as a provisional method for this association, but am not certain. Perhaps someone can give me some definite information on the subject.

The PRESIDENT. It seems to me that the Schulze-Tiemann method should be given a trial by the reporter next year.

Mr. KILGORE. The Schulze-Tiemann method was adopted as a provisional method two years ago, and, having been so adopted, it may come properly before the association for adoption as a regular method.

Mr. WINTON. I would like an expression of opinion in regard to the Schulze-Tiemann method. How many present have used this method? Several members responded affirmatively.

Mr. STREET. I would gladly include the Schulze-Tiemann method among my recommendations as one which should be further tried and possibly adopted by the association.

The PRESIDENT. By common consent the Schulze-Tiemann method will be considered by the committee on recommendations of reporters.

Mr. HUSTON. I would like to know if it be necessary, as directed in the methods of analysis, to use a flat-bottomed flask in nitrogen determinations by the Ulsch-Street method.

Mr. STREET. It is not necessary; it is only a matter of convenience.

Mr. HUSTON. Sometimes mere details of description which are not important become official, and the analyst feels compelled to follow even the minutiae of the description, when any little variation therefrom would be of no importance, and often prove a great convenience.

Mr. KILGORE. I suggest that Mr. Winton furnish the committee on recommendations of reporters a description of the Schulze-Tiemann method.

Mr. GAINES. I have had considerable trouble with the breaking of flasks during distillation. About one-half the flasks, in my experience, break in distilling. I should like to know how this breakage can be avoided. It is a difficulty I am puzzled to account for.

Mr. GOSS. In the determination of the nitrogen in soils the size of the flask should be taken into consideration. If the flask is large enough there will be very little trouble with bumping, and the bumping in the flask is usually the cause of the breakage.

Mr. STREET. It is also important to get the right kind of flasks. If you do this you will have very little trouble by breaking. In making 400 analyses I have only broken six flasks.

Mr. GAINES. May I ask Mr. Street if he transfers the material from the digesting flask to a separate flask for distillation?

Mr. STREET. We never transfer from one flask to another. We make the distillation in the same flask in which the digestion in sulphuric acid is accomplished.

Mr. VAN SLYKE. I use the Jena glassware. While it is more expensive at first, I find it cheaper in the end, and it gives most excellent satisfaction, and the amount of breakage is reduced to the minimum.

The PRESIDENT. When a little zinc dust is used during the distillation I have found very little trouble from the breaking of flasks.

Mr. WHEELER. Often the addition of a little more caustic soda will avoid bumping and consequent breaking of the flask.

The PRESIDENT. If there is no further discussion of the report on nitrogen the recommendations therein made will be referred to the committee on recommendations of reporters for their consideration. I will now call for the report on potash.

Mr. WINTON. Mr. President, I beg leave to submit the following report:

REPORT ON POTASH.

By A. L. WINTON, *Reporter*.

The reporter on potash for the years 1894 and 1895, Mr. H. J. Wheeler, realized the importance of learning more with regard to our old methods before taking up the various new methods which had not yet been tried by the association, and accordingly confined the work carried on during the two years to a thorough investigation of the Lindo-Gladding and "optional" methods as applied to mixed fertilizers. Since in this country the larger part of the potash which is sold to the farmer is contained in mixed fertilizers, it was desirable that our methods for the analysis of these goods be first tested.

This work accomplished it was thought proper during the present year to carry on a similar investigation with German potash salts.

Incidentally a study was made of factors for the conversion of potassium platinichlorid into potassium sulphate, chlorid, and oxid.

INVESTIGATION OF METHODS FOR DETERMINATION OF POTASH IN GERMAN POTASH SALTS.

Although, as has been stated, the potash applied to our soils is largely contained in mixed fertilizers, still it is practically all brought into the country in the form of four German potash salts. It is then on the salts themselves that the analyses of European and American chemists are most frequently compared, and any discrepancies in the results are too often attributed to inaccuracies in our methods.

Before going into the details of the work carried on during the year, it may be well to consider the principles which underlie the "optional," Stassfurt, and Lindo-Gladding methods.

Our so-called "optional method" is really but a modification of a method which in one form and another has for many years been employed in general analytical chemistry. The salient feature is the separation of sulphuric acid and all bases but potash and soda previous to evaporation with platinum solution. This necessitates several precipitations, the evaporation of a considerable bulk of liquid and ignition for removal of ammonium salts, thus making the process a very tedious one. Since barium sulphate is apt to carry down with it a considerable amount of potash, the results are usually low.

The "shorter method" devised by Fresenius¹ and in a slightly modified form² employed under the name of the "Stassfurt method" in the laboratories of the

¹ Quant. Analyse, 6 Aufl. II, 292.

² Ztschr. Angew. Chem., 1895.

Stassfurt syndicate and the German experiment stations, is really a simplification of the foregoing method. It depends on the fact that if sulphuric acid be removed from the solution of a potash salt, the liquid may be evaporated directly with platinum solution, chlorids of magnesium, calcium and sodium forming alcohol-soluble salts with chloroplatinic acid. It is claimed that if the precipitation of barium sulphate be carried on in an acid solution and with certain precautions, loss of potash is avoided.

The Lindo-Gladding method has been developed by the successive labors of Finkener,¹ Lindo,² Gladding,³ and members of this association. The solution in which potash is to be determined is evaporated with platinum solution without previous separation of bases or sulphuric acid, the sulphates which contaminate the potassium platinichlorid being subsequently removed by ammonium chlorid solution. The method is rapid and, as will be seen later, remarkably accurate.

During the year only the two methods recognized by the association have been studied by American chemists, but in three of the laboratories connected with the Stassfurt industry, comparative analyses were made by the Lindo-Gladding and Stassfurt methods.

PLAN OF INVESTIGATION.

Instead of working with the German salts themselves, the potash content of which could only be learned by recourse to the very methods which it sought to test, or with artificial German salts containing a known proportion of potash from pure salts, which, however, are liable to lack uniformity and change in composition through loss of moisture, the plan followed by Mr. Wheeler was adopted which obviates all these disadvantages.

In each determination a carefully weighed portion of chemically pure potash salt was employed and to this was added a solution of the other water-soluble ingredients in the proportion contained in the material, the analysis of which it was desired to imitate. Such a solution contains the same ingredients as a solution of the commercial potash salt, but the exact quantity of potash present is definitely known without analysis.

PREPARATION OF MATERIALS.

The statements of the average composition of the Stassfurt salts, given in a pamphlet issued by the German Kali Works, served as a basis for the preparation of the solutions of analysis representing these salts. (See Table I.)

TABLE I.—*Average composition of German potash salts.*

[Taken from "Potash; origin, trade and its use in agriculture," Columbian Exposition, Chicago, German Kali Works.]

	K ₂ SO ₄ .	KCl.	MgSO ₄ .	MgCl ₂ .	NaCl.	CaSO ₄ .	Sub- stances insoluble in water.	Water.	K ₂ O.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Kainit.....	21.3	2.0	14.5	12.4	34.6	1.7	0.8	12.7	12.8
Sulphate of potash, 90 per cent.	90.6	1.6	2.7	1.0	1.2	0.4	0.3	2.2	49.9
Sulphate of potash—magnesia	50.4	34.0	2.5	0.9	0.6	11.6	27.2
Muriate of potash, 80 to 85 per cent.....	83.5	0.4	0.3	14.5	0.2	1.1	52.7

Both potassium chlorid and sulphate were used in the work. The former was from the same lot as that employed in last year's work, and contained 0.45 per cent

¹ Pogg. Ann. 29, 627.

² Chem. News, 44, pp. 77, 86, 97 and 129.

³ U. S. Department of Agriculture, Bulletin No. 7, p. 38.

of water, which being included in the crystals could only be determined by heating nearly to redness in a well-covered crucible. Included water was also contained in the potassium sulphate when received from the dealer, but in this case the whole quantity was ignited by the reporter until entirely anhydrous. There was every evidence that both salts were absolutely pure.

The other salts used were chemically pure with one exception. The reporter found that "magnesium chlorid chemically pure," from no less than four manufacturers, contained so considerable a quantity of potash as to be valueless for the work. After some search a cheaper grade of the salt was obtained, which, although not claimed to be pure, was almost entirely free from potash, and therefore better suited for the purpose than the so-called chemically pure salts. It contained the normal quantity of water of crystallization—6 molecules.

The magnesium sulphate was recrystallized Epsom salts with 7 molecules of water. The sodium chlorid and calcium sulphate were not only pure but anhydrous.

Four mixtures of impurities representing the water-soluble ingredients, other than potash, of the four manure salts were prepared by mixing the salts just named in the proportions given in Table 1, allowing for water of crystallization when present. These mixtures were then weighed into portions, representing in each case the impurities in 10 grams of the manure salt, and bottled.

A bottle of each of these, together with portions of the pure potash salts, were sent out to each chemist with the following instructions:

INSTRUCTIONS FOR THE WORK.

Determinations of potash should first be made on the pure potash salts, using tested reagents, and the results compared with the theoretical composition. The total quantity of each of the mixtures of impurities should be boiled with 200 to 300 cc of water, made up to 500 cc and filtered through a dry paper. Solutions for analysis, representing 0.5 gram of the several Stassfurt products are prepared as follows:

Kainit: Dissolve about 0.14 gram K_2SO_4 in 25 cc kainit impurities.

Sulphate potash, 90 per cent: Dissolve about 0.46 gram K_2SO_4 in 25 cc sulphate potash.

Sulphate potash and magnesia: Dissolve about 0.26 gram K_2SO_4 in 25 cc sulphate potash and magnesia.

Muriate potash, 80 per cent: Dissolve about 0.42 gram KCl in 25 cc muriate of potash.

The pure potash salts should be weighed with the greatest accuracy from weighing tubes, taking care to have the quantities within a few centigrams of those given above—better more than less. Solutions thus prepared should be used in testing the alternate method and also in testing the Lindo-Gladding method, as applied to all the salts except kainit. In the analysis of this latter salt by the official Lindo-Gladding method lime is first separated, although in the original method as described by Gladding and followed for several years by the association, the evaporation with platinum solution was made directly. De Roode (*J. Am. Chem. Soc.* 17, 85) finds separation of lime unnecessary.

You are requested first to follow the same method for kainit as for sulphate of potash and magnesia, on solutions prepared as described, then, if desirable, make determinations with separation of lime as follows: Place a carefully weighed portion of about 0.28 gram K_2SO_4 and 50 cc kainit impurities in a 100 cc flask, add ammonia and ammonium oxalate, make up to 100 cc, filter through a dry paper, and proceed with 50 cc according to the official method for kainit.

If you undertake a study of the Stassfurt methods (*Ztschr. Angew. Chem.*, 1895, Heft 17), it is recommended, in order to avoid waste of material, that the pure salt and solution of impurities be taken in each case in such quantities as to correspond to one-fifth (or for kainit, method C. 2. to one-twentieth) the quantity of the Stassfurt product which in loc. cit. it is directed should be weighed out, using correspondingly smaller quantities of reagents and making up to a correspondingly smaller volume. In this way the aliquot portion after separation of $BaSO_4$ may be made to represent the same quantity of material as it would if the directions were strictly followed.

Blank determinations should be made by the methods investigated on the solution of impurities, using the same quantities of reagents as in the actual analysis.

In reporting results please give (1) weight of pure salt employed, (2) total weight K_2PtCl_6 found, and (3) weight of K_2PtCl_6 from reagents and "impurities."

Twenty-six sets of materials were sent to American chemists who expressed an interest in the work. Of these seven found opportunity for conducting the analyses.

At the suggestion of Dr. von Herff, ten sets were sent to the New York office of the Kali Syndicate from whence six were forwarded to German experiment stations and four to laboratories connected with the Stassfurt industry.

No communication has been received from the German stations, but Dr. Tietjens reports results obtained in the laboratory of the syndicate and at two of the salt mines.

RESULTS SECURED BY AMERICAN CHEMISTS.

The following chemists took part in the study of the Lindo-Gladding and "optional" methods:

Mr. B. B. Ross, State chemist, Auburn, Ala.

Mr. F. B. Bomberger, laboratory of State chemist, Maryland Agricultural College, College Park, Md.

Mr. W. G. Brown, United States Department of Agriculture, Washington, D. C.

Mr. C. G. Hopkins, Illinois Experiment Station, Urbana, Ill.

Mr. G. W. Cavanaugh, Cornell Experiment Station, Ithaca, N. Y.

Mr. C. H. Jones, Vermont Experiment Station, Burlington, Vt.

Mr. F. S. Shiver, Clemson Experiment Station, Clemson College, S. C.

In Table II are given the weights of the pure salts taken, of the total K_2PtCl_6 found and of the K_2PtCl_6 from "impurities" and reagents. These figures furnish the data for the calculation given in the subsequent tables. It should be noted that although the impurities were almost entirely free from potash, several of the analysts obtained K_2PtCl_6 in blank determinations on the reagents and impurities, which was probably due to potash in the reagents. This was particularly marked when the optional method was followed. This same experience was encountered in last year's work, and illustrates the importance of testing reagents by blank determinations, since "chemically pure" salts when carefully examined are often found to contain impurities.

TABLE II.—*Analytical data from American chemists.*

	Lindo-Gladding method.			Optional method.		
	Pure salt taken.	K_2PtCl_6 total found.	K_2PtCl_6 reagents and impurities.	Pure salt taken.	K_2PtCl_6 total found.	K_2PtCl_6 reagents and impurities.
Potassium chlorid c. p. :	Gram.	Gram.	Gram.	Gram.	Gram.	Gram.
F. B. Bomberger	0.2500	0.8141	0.0035
	.2500	.8158
	.2500	.8168
W. G. Brown4586	1.4882	.0003	¹ 0.3998	² 1.3027	0.0003
	.6824	2.2169	² .4836	1.5676
	.4653	1.5100	² .4343	1.4074
	.3959	1.29414569	1.4782
4508	1.4487
G. W. Cavanaugh4821	1.5720
3947	1.2908
C. H. Jones5000	1.6265	.0025	.5000	1.6240	.0070
	.5000	1.62955000	1.6214
	.5000	1.6295
F. S. Shiver	² .1671	.5433	.0000
A. L. Winton	² .6096	1.9793	.0000
	² .5400	1.7512
Muriate of potash (KCl c. p. + impurities):						
B. B. Ross	¹ .2100	.6930	.0068
	¹ .2100	.6916

¹ Anhydrous salt.

² Direct evaporation with platinum solution.

TABLE II.—*Analytical data from American chemists—Continued.*

	Lindo-Gladding method.			Optional method.		
	Pure salt taken.	K ₂ PtCl ₆ total found.	K ₂ PtCl ₆ reagents and impurities.	Pure salt taken.	K ₂ PtCl ₆ total found.	K ₂ PtCl ₆ reagents and impurities.
Muriate of potash (KCl c. p. + impurities)—Continued.	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>
F. B. Bomberger4516	1.4712	.0065	.1563	.5197	.0163
	.4706	1.53151563	.5182
W. G. Brown.....	.4059	1.3226	.0001	.4310	1.3937	.0010
	.5036	1.63794814	1.5508	.0005
G. W. Cavanaugh.....	.4439	1.45354228	1.3806	.0027
C. G. Hopkins.....	.4555	1.4802	.0005	¹ .4206	1.3600	.0013
	.4318	1.4020	.0005	¹ .4205	1.3602	.0007
C. H. Jones.....	.4215	1.3754	.0045	.4200	1.3740	.0100
	.4220	1.37654200	1.3718
F. S. Shiver.....	.4261	1.3822	.0001	.4214	1.3815	.0062
A. L. Winton4290	1.3915	.0003
	.3948	1.2780
Potassium sulphate c. p. :						
F. B. Bomberger2500	.7011	.0035	.1310	.3804	.0173
	.2500	.70021310	.3803
	.2500	.6995
W. G. Brown5649	1.5795	.0003	.5121	1.4080	.0003
	.6011	1.67944822	1.3227
G. W. Cavanaugh.....	.4836	1.3566
	.4842	1.3565
C. H. Jones5000	1.3987	.0025	.5000	1.3915	.0075
	.5000	1.39905000	1.3940
F. S. Shiver.....1643	.4589	.0000
A. L. Winton	² .4725	1.3223	.0000
	² .4942	1.3786
	² .4185	1.1700
	² .4679	1.3064
Sulphate of potash, 90 per cent (K₂SO₄ c. p. + impurities):						
F. B. Bomberger3930	1.1002	.0049	.1310	.3814	.0187
	.4092	1.13891310	.3818
	.4624	1.2917
W. G. Brown.....	.5130	1.4337	.0003	.6171	1.6898	.0020
	.4517	1.26324371	1.2124	.0025
G. W. Cavanaugh.....	.4745	1.3319	.0039	.4506	1.2469
C. G. Hopkins.....	.4674	1.3055	.0008	¹ .4604	1.2744	.0013
	.4563	1.2730	.0008	¹ .4603	1.2700	.0013
C. H. Jones4640	1.2980	.0040	.4600	1.2810	.0095
	.4645	1.29604600	1.2835
A. L. Winton4042	1.1248	.0005
	.4121	1.1483
	.4110	1.1448
Sulphate of potash and magnesia (K₂SO₄ + impurities):						
F. B. Bomberger2670	.7494	.0055	.1310	.3782	.0180
	.2785	.78081310	.3797
1310	.3780

¹ See Mr. Hopkins's notes.² Direct evaporation with platinum solution.

TABLE II.—Analytical data from American Chemists—Continued.

	Lindo-Gladding method.			Optional method.		
	Pure salt taken.	K ₂ PtCl ₆ total found.	K ₂ PtCl ₆ reagents and impurities.	Pure salt taken.	K ₂ PtCl ₆ total found.	K ₂ PtCl ₆ reagents and impurities.
Sulphate of potash and magnesia (K ₂ SO ₄ + impurities)—Continued.	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>
W. G. Brown2680	.7508	.0021	.2676	.7318	.0033
	.3301	.92522509	.6854	.0027
G. W. Cavanaugh.....				.2652	.7371
C. G. Hopkins.....	.2617	.7302	.0007	¹ .2610	.7295	.0017
	.2553	.7124	.0007	¹ .2604	.7260	.0017
C. H. Jones2645	.7434	.0035	.2600	.7240	.0090
	.2640	.74002600	.7248
F. S. Shiver.....	.2650	.7358	.0000	.2632	.7374	.0151
A. L. Winton3040	.8470	.0009
	.2686	.7490
	.2566	.7136
Kainit (K ₂ SO ₄ c. p. + impurities):						
B. B. Ross.....	² .1400	.3950	.0044
	² .1400	.3941	.0044
F. B. Bomberger1390	.3898	.0040	.1310	.3798	.0153
	.1310	.36841310	.3801
	.1310	.3674
W. G. Brown.....	² .2009	.5624	.0034	.3700	1.0217	.0028
	² .1555	.43771395	.3889	.0047
	.1460	.4070	³ .4180	1.3394
	.1871	.5190	³ .4427	1.4416
G. W. Cavanaugh.....	.1464	.4004	.0044	.1543	.4287
C. G. Hopkins1518	.4226	.0007	¹ .1405	.3946	.0021
	.1571	.4381	.0006	¹ .1407	.3946	.0025
C. H. Jones.....	² .1460	.4090	.0030	.1400	.3962	.0105
	² .1460	.40801400	.4010
	.1445	.4022	.0035
	.1445	.4080
F. S. Shiver.....	.1433	.3968	.0000	.1444	.4081	.0097
A. L. Winton	² .1387	.3860	.0013
	² .1727	.4807

¹ See Mr. Hopkins's notes.² Direct evaporation without previous separation of lime.³ Potassium chlorid.

Mr. Ross sent the following notes with his figures:

The Lindo-Gladding process, as originally employed by the association, was used as regards the preliminary treatment of the solution without any attempt being made to precipitate the lime, etc., by addition of NH₄HO and (NH₄)₂C₂O₄.

After weighing the precipitates they were dissolved off the filter with hot water and the loss on reweighing noted. The proportion of impurities left undissolved by the NH₄Cl wash solution was quite small, but for accuracy's sake the loss on dissolving in hot H₂O was noted. In several cases this aqueous solution of K₂PtCl₆ was evaporated on a water bath and the residue dried. When dried at 100° C. the residue invariably weighed more than the loss in weight noted on dissolving the contents of the filter in hot H₂O. On drying at 120° to 130° C. the excess in this weight soon disappeared.

Although I have made quite a number of determinations in two or three of the salts by the alternate method, I do not feel justified in reporting some of these (which are too low in almost every case), for the reason that I had numerous interruptions during this part of the work. My experience with the alternate method has always been that it is difficult to so adjust the flame used in the final ignition of

the residue (obtained by evaporation) as to secure complete volatilization of ammonium compounds and at the same time to avoid loss of KCl.

Mr. Brown wrote as follows:

I have followed your instructions as far as they were given, except where otherwise noted, but in the matter of detail I may have done differently from some of the other workers. To explain: In evaporating the solution of the potassium salt after the addition of the platinum chlorid I have been careful to get rid of the excess of hydrochloric acid, in some cases adding water and evaporating two or three times until the odor of hydrochloric acid was hardly perceptible. At present there are no directions for the treatment of the dried mass with alcohol. I added the alcohol to the mass and kept it only so long in contact with the salts as it seemed to be dissolving them, pouring off the solution through the Gooch crucible or filter, adding fresh alcohol and continuing to treat with alcohol until it came away nearly colorless before transferring the potassium chlorplatinat to the Gooch crucible or filter. In five minutes after adding the alcohol it was frequently decanted off. I found it necessary to use about 100 cc of alcohol for each determination, usually somewhat more than less. The potassium chlorplatinat was in all cases dried in the steam bath at 100° (the temperature is not given in the "Methods of analysis" for the Lindo-Gladding method), except in two experiments, where, after having been dried in the steam bath, it was treated in the air bath at 130° for about three hours, when it was found that the weight was unchanged. In two of the determinations by the optional method there were added to the potassium chlorid kainit impurities, instead of muriate of potash impurities, and the determination of potassium chlorplatinat was made under these conditions.

In one of the sulphate of potash determinations by the optional method instead of 0.46 gram potassium sulphate 0.62 gram was taken and 50 cc of impurities.

In all the determinations except two by the optional method the potassium chlorplatinat was weighed in Gooch crucibles. In these two the potassium chlorplatinat was collected on paper, dissolved off the paper with hot water, caught in a platinum dish, evaporated, dried, and weighed in the dish.

Mr. Hopkins stated that—

In the optional method where heavy precipitates were formed by adding BaCl and Ba(OH)₂ they were washed twice by ordinary decantation. Then they were rubbed up with about 5 grams of purified sand and shaken well with hot water, which was decanted after the precipitate settled. This was continued until washings free from chlorids were obtained.

The remainder of the figures were reported without comment, other than those given with the tables.

TABLE III.—*Results from American chemists corrected for potash in reagents and "impurities," calculated by old factors.*

	Lindo-Gladding method.				Optional method.			
	Taken KCl.	Found KCl.	Error KCl.	Error K ₂ O.	Taken KCl.	Found KCl.	Error KCl.	Error K ₂ O.
Potassium chlorid c. p.:	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>
F. B. Bomberger	0.2489	0.2477	—0.0012	—0.0008
	.2489	.2482	— .0007	— .0004
	.2489	.2485	— .0004	— .0003
W. G. Brown.....	.4565	.4547	— .0018	— .0011	¹ 0.3998	0.3980	—0.0018	—0.0011
	.6793	.6774	— .0019	— .0012	¹ .4814	.4790	— .0024	— .0015
	.4632	.4614	— .0018	— .0011	¹ .4324	.4300	— .0024	— .0015
	.3941	.3954	+ .0013	+ .0008	.4548	.4516	— .0032	— .0020
4488	.4426	— .0062	— .0039
G. W. Cavanaugh4799	² .4804	+ .0005	+ .0003
3930	² .3944	+ .0014	+ .0009
C. H. Jones.....	.4978	.4963	— .0015	— .0010	.4978	.4942	— .0036	— .0023
	.4978	.4972	— .0006	— .0004	.4978	.4934	— .0044	— .0028
	.4978	.4972	— .0006	— .0004

¹ Direct evaporation with platinum solution.

² No blank determinations made, hence results not corrected.

TABLE III.—Results from American chemists corrected for potash in reagents and “impurities,” calculated by old factors—Continued.

	Lindo-Gladding method.				Optional method.			
	Taken KCl.	Found KCl.	Error KCl.	Error K ₂ O.	Taken KCl.	Found KCl.	Error KCl.	Error K ₂ O.
Potassium chlorid c. p.—Continued.	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>
F. S. Shiver	¹ . 1664	.1660	— .0004	— .0002
A. L. Winton.....	¹ . 6069	.6049	— .0020	— .0013
.....	¹ . 5376	.5352	— .0024	— .0015
Muriate of potash, 80 to 85 per cent (KCl c. p. + impurities):								
B. B. Ross.....	.2100	.2097	— .0003	— .0002
.....	.2100	.2093	— .0007	— .0004
F. B. Bomberger4496	.4476	— .0020	— .0013	.1556	.1537	— .0019	— .0012
.....	.4685	.4660	— .0025	— .0016	.1536	.1532	— .0024	— .0015
W. G. Brown.....	.4041	.4042	+ .0001	+ .0001	.4291	.4257	— .0034	— .0021
.....	.5013	.5005	— .0008	— .0005	.4792	.4737	— .0055	— .0035
G. W. Cavanaugh4419	² . 4442	+ .0023	+ .0014	.4209	.4211	+ .0002	+ .0001
C. G. Hopkins4534	.4522	— .0012	— .0008	³ . 4187	.4153	— .0034	— .0021
.....	.4299	.4283	— .0016	— .0010	³ . 4186	.4154	— .0032	— .0020
C. H. Jones.....	.4196	.4189	— .0007	— .0004	.4181	.4168	— .0013	— .0008
.....	.4201	.4193	— .0008	— .0005	.4181	.4162	— .0019	— .0012
F. S. Shiver4242	.4224	— .0018	— .0011	.4195	.4203	+ .0008	+ .0005
A. L. Winton4271	.4251	— .0020	— .0013
.....	.3930	.3905	— .0025	— .0016

	Lindo-Gladding method.				Optional method.			
	Taken K ₂ SO ₄ .	Found K ₂ SO ₄ .	Error K ₂ SO ₄ .	Error K ₂ O.	Taken K ₂ SO ₄ .	Found K ₂ SO ₄ .	Error K ₂ SO ₄ .	Error K ₂ O.
Potassium sulphate c. p.:	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>
F. B. Bomberger.....	0.2500	0.2490	—0.0010	—0.0005	0.1310	0.1296	—0.0014	—0.0008
.....	.2500	.2487	— .0013	— .0007	.1310	.1296	— .0014	— .0008
.....	.2500	.2485	— .0015	— .0008
W. G. Brown.....	.5649	.5638	— .0011	— .0006	.5121	.5025	— .0096	— .0051
.....	.6011	.5994	— .0017	— .0009	.4822	.4721	— .0101	— .0054
G. W. Cavanaugh4836	² . 4843	+ .0007	+ .0004
.....	.4842	² . 4843	+ .0001	+ .0001
C. H. Jones.....	.5000	.4984	— .0016	— .0009	.5000	.4941	— .0059	— .0032
.....	.5000	.4986	— .0014	— .0008	.5000	.4950	— .0050	— .0027
F. S. Shiver1643	.1638	— .0005	— .0003
A. L. Winton.....	¹ . 4725	.4720	— .0005	— .0003
.....	¹ . 4942	.4922	— .0020	— .0011
.....	¹ . 4185	.4177	— .0008	— .0004
.....	¹ . 4679	.4664	— .0015	— .0008
Sulphate of potash, 90 per cent (K ₂ SO ₄ c. p. + impurities):								
F. B. Bomberger.....	.3930	.3910	— .0020	— .0011	.1310	.1295	— .0015	— .0008
.....	.4092	.4048	— .0044	— .0024	.1310	.1296	— .0014	— .0007
.....	.4624	.4594	— .0030	— .0016
W. G. Brown5130	.5117	— .0013	— .0007	.6171	.6024	— .0147	— .0089
.....	.4517	.4509	— .0008	— .0004	.4371	.4320	— .0051	— .0028
G. W. Cavanaugh4745	.4741	— .0004	— .0002	² . 4506	.4452	— .0054	— .0029

¹ Direct evaporation with platinum solution.² No blank determinations made, hence results not corrected.³ See Mr. Hopkins's notes.

TABLE III.—*Results from American chemists corrected for potash in reagents and "impurities," calculated by old factors—Continued.*

	Lindo-Gladding method.				Optional method.			
	Taken K ₂ SO ₄ .	Found K ₂ SO ₄ .	Error K ₂ SO ₄ .	Error K ₂ O.	Taken K ₂ SO ₄ .	Found K ₂ SO ₄ .	Error K ₂ SO ₄ .	Error K ₂ O.
Sulphate of potash, 90 per cent (K ₂ SO ₄ c. p. + impurities)— Continued.	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>
C. G. Hopkins4674	.4658	— .0016	— .0009	¹ .4604	.4545	— .0059	— .0032
	.4563	.4542	— .0021	— .0011	¹ .4603	.4529	— .0074	— .0040
C. H. Jones.....	.4640	.4620	— .0020	— .0011	.4600	.4539	— .0061	— .0033
	.4645	.4612	— .0033	— .0018	.4600	.4548	— .0052	— .0028
A. L. Winton.....	.4042	.4014	— .0028	— .0015
	.4121	.4098	— .0023	— .0012
	.4110	.4085	— .0025	— .0014
Sulphate of potash and mag- nesia (K ₂ SO ₄ c. p. + impuri- ties):								
F. B. Bomberger.....	.2670	.2656	— .0014	— .0008	.1310	.1286	— .0024	— .0013
	.2785	.2768	— .0017	— .0009	.1310	.1291	— .0019	— .0010
1310	.1285	— .0025	— .0014
W. G. Brown.....	.2680	.2673	— .0007	— .0004	.2676	.2602	— .0074	— .0040
	.3301	.3295	— .0006	— .0003	.2509	.2436	— .0073	— .0039
G. W. Cavanaugh.....	² .2652	.2631	— .0021	— .0012
C. G. Hopkins2617	.2604	— .0013	— .0007	¹ .2610	.2598	— .0012	— .0006
	.2553	.2541	— .0012	— .0006	¹ .2604	.2586	— .0018	— .0010
C. H. Jones.....	.2645	.2642	— .0003	— .0002	.2600	.2553	— .0047	— .0025
	.2640	.2629	— .0011	— .0006	.2600	.2555	— .0045	— .0024
F. S. Shiver.....	.2650	.2627	— .0023	— .0012	.2632	.2579	— .0053	— .0029
A. L. Winton.....	.3040	.3021	— .0019	— .0010
	.2686	.2671	— .0015	— .0008
	.2566	.2544	— .0022	— .0012
Kainit (K ₂ SO ₄ c. p. + impuri- ties):								
B. B. Ross	³ .1400	.1394	— .0006	— .0003
	³ .1400	.1391	— .0009	— .0005
F. B. Bomberger.....	.1390	.1377	— .0013	— .0007	.1310	.1301	— .0009	— .0005
	.1310	.1301	— .0009	— .0005	.1310	.1302	— .0008	— .0004
	.1310	.1297	— .0013	— .0007
W. G. Brown.....	³ .2009	.1996	— .0013	— .0007	.3700	.3634	— .0066	— .0036
	³ .1555	.1550	— .0005	— .0003	.1395	.1375	— .0020	— .0011
	.1460	.1441	— .0019	— .0010	⁴ .4161	.4082	— .0079	— .0043
	.1871	.1841	— .0030	— .0016	⁴ .4407	.4394	— .0013	— .0007
G. W. Cavanaugh1464	.1414	— .0050	— .0027	² .1543	.1530	— .0013	— .0007
C. G. Hopkins1518	.1507	— .0011	— .0006	¹ .1405	.1400	— .0005	— .0003
	.1571	.1562	— .0009	— .0005	¹ .1407	.1400	— .0007	— .0004
C. H. Jones.....	³ .1460	.1449	— .0011	— .0006	.1400	.1377	— .0023	— .0012
	³ .1460	.1446	— .0014	— .0008	.1400	.1394	— .0006	— .0003
	.1445	.1423	— .0022	— .0012
	.1445	.1444	— .0001	— .0001
F. S. Shiver.....	.1433	.1417	— .0016	— .0009	.1444	.1422	— .0022	— .0012
A. L. Winton.....	³ .1387	.1373	— .0014	— .0008
	³ .1727	.1711	— .0016	— .0009

¹ See Mr. Hopkins's notes.² No blank determinations made, hence results not corrected.³ Direct evaporation, without previous separation of lime.⁴ Potassium chloride.

The figures in Table III are calculated from the analytical data of Table II. The weights of KCl taken are corrected for the moisture contained in the salt. This was not necessary in the case of the K_2SO_4 , as the salt was anhydrous. The weights of KCl and K_2SO_4 found were obtained by deducting from the total K_2PtCl_6 that found in the reagents and impurities and multiplying by the commonly used factors 0.3056 for KCl and 0.357 for K_2SO_4 . Mr. Cavanaugh failed to make blank determinations in a number of cases, and these results, not being corrected, are high.

It should be borne in mind that the quantities used in the case of the artificial German salts usually represent approximately 0.5 of the real salt, and therefore the errors given in grams in the table may be readily converted into percentage errors by moving the decimal point two places to the right and multiplying by two.

TABLE IV.—Difference between K_2O taken and K_2O found, calculated by old and new factors—Average results of American chemists.

[Difference given in milligrams.]

Method.	B. B. Ross.		F. B. Bomberger.		W. G. Brown.		G. W. Cavanaugh.		C. G. Hopkins.	
	Old factor.	New factor.	Old factor.	New factor.	Old factor.	New factor.	Old factor.	New factor.	Old factor.	New factor.
Direct evaporation, no $(NH_4)Cl$:										
Potassium chlorid c. p.					-1.4	-0.3				
Potassium sulphate c. p.										
Lindo-Gladding method:										
Potassium chlorid c. p.			-0.5	+0.2	-1.1	+0.2				
Muriate of potash	-0.3	+0.2	-1.5	-0.2	-0.2	+1.0			-0.9	+0.2
Potassium sulphate c. p.			-0.7	-0.1	-0.7	+0.7				
Sulphate of potash, 90 per cent.			-1.7	-0.8	-0.5	+0.6	-0.2	+0.9	-1.0	+0.1
Sulphate of potash and magnesia.			-0.8	-0.1	-0.3	+0.4			-0.6	0.0
Kainit (lime not separated)	-0.4	0.0			-0.5	0.0				
Kainit (lime separated)			-0.6	-0.3	-1.3	-0.9	-2.7	-2.3	-0.5	-0.1
Optional method:										
Potassium chlorid c. p.					-2.9	-1.7				
Muriate of potash			-1.3	-0.9	-2.8	-1.6	+0.1	+1.3	-2.0	-1.0
Potassium sulphate c. p.			-0.8	-0.4	-5.2	-4.0				
Sulphate of potash, 90 per cent.			-0.7	-0.4	-5.8	-4.0			-3.6	-2.4
Sulphate of potash and magnesia.			-1.2	-0.9	-3.9	-3.3			-0.8	-0.1
Kainit			-0.4	-0.1	-2.3	-1.7			-0.3	0.0

Method.	C. H. Jones.		F. S. Shiver.		A. L. Winton.		Average.	
	Old factor.	New factor.	Old factor.	New factor.	Old factor.	New factor.	Old factor.	New factor.
Direct evaporation, no $(NH_4)Cl$:								
Potassium chlorid c. p.			-0.2	+0.2	-1.4	+0.2	-1.0	0.0
Potassium sulphate c. p.					-0.6	+0.5	-0.6	+0.5
Lindo-Gladding method:								
Potassium chlorid c. p.	-0.6	+0.7					-0.7	+0.4
Muriate of potash	-0.4	+0.6	-1.1	-0.1	-1.4	-0.3	-0.8	+0.2
Potassium sulphate c. p.	-0.8	+0.4					-0.7	+0.3
Sulphate of potash, 90 per cent.	-1.4	-0.3			-1.4	-0.3	-1.0	0.0
Sulphate of potash and magnesia.	-0.4	+0.3	-1.2	-0.6	-1.0	-0.2	-0.7	0.0
Kainit (lime not separated)	-0.7	-0.3			-0.8	-0.4	-0.6	-0.2
Kainit (lime separated)	-0.6	-0.3	-0.9	-0.5			-1.1	0.0
Optional method:								
Potassium chlorid c. p.	-2.5	-1.2					-2.7	-1.4
Muriate of potash	-1.0	+0.1	+0.5	+1.6			-1.1	-0.2
Potassium sulphate c. p.	-2.9	-1.7	-0.3	+0.2			-2.3	-1.5
Sulphate of potash, 90 per cent.	-3.0	-1.9					-3.3	-2.2
Sulphate of potash and magnesia.	-2.4	-1.8	-2.9	-2.2			-2.2	-1.7
Kainit	-0.7	-0.4	-1.2	-0.8			-1.0	-0.6

Table IV shows the errors expressed in milligrams of K_2O , of the average results of each analyst calculated both by the old factors and by those based on revised atomic weights. It will be seen from Tables III and IV that as a rule considerable potash was lost in carrying out the optional method. This is due to an inherent defect of the process. It has long been known that barium sulphate carries down potash with it and the work of the past three years furnishes abundant illustration of this fact. The errors introduced, especially in the presence of sulphates, are so considerable as to render this method very inaccurate, and if it can not be so modified as to remedy this defect it should be stricken from our list.

It might be well to investigate, with regard to this point, the plan for precipitation followed by the Stassfurt chemists, namely, addition of barium chlorid, dropwise, to a strongly acid and boiling solution.

The results of the different analysts by the Lindo-Gladding method stand out in striking contrast to those by the optional method, being in nearly every case concordant, and, by the old factors, a trifle below theory. The slight minus errors, as will be discussed later, disappear almost entirely if the factors based on the universally accepted atomic weight of platinum be employed. Notwithstanding the criticisms of Breyer and Schweitzer, the Stassfurt chemists, Vogel and Haefcke, *the results of eight chemists working independently of each other in different laboratories and with different reagents, agree in establishing beyond a reasonable doubt, the remarkable accuracy of the Lindo-Gladding method.*

An experienced analyst who follows strictly the instructions and uses a Gooch crucible, should find it rapid, economical, and accurate.

It may be that the method has not given good results in the hands of European chemists, because the potassium platinichlorid has been filtered and weighed in some other way than on a Gooch crucible, which is universally employed in experiment stations and fertilizer control laboratories in this country.

If ammonium chlorid solution be poured through the double salt contained on an ordinary filter paper double decomposition may take place, the impurities may not be washed out thoroughly and the difficulty in removing the ammonium chlorid with alcohol may be encountered.

While the use of the Gooch crucible has been explicitly directed for the optional method, it should be specified also among the details for carrying out the Lindo-Gladding method.

It was used in obtaining all the results by American chemists given in the last three reports (except in a few cases otherwise noted) and undoubtedly in the work of preceding years.

Breyer and Schweitzer, and Vogel and Haefcke¹ claim that the removal of impurities from potassium platinichlorid is inadmissible, since thereby, through double decomposition, ammonium platinichlorid and potassium chlorid are formed, the former remaining as an impurity on the filter, the latter being carried into the filtrate. Fin Rener² first pointed out that this reaction takes place, but his results show that the change is very gradual. He found that a saturated solution of ammonium chlorid at 22° C., when in contact with finely divided potassium platinichlorid for one hour, decomposed but 0.27 per cent of the latter. Since our methods secure the double salt in a granular condition, which according to Fin Rener is not so rapidly acted on, and since operating with a Gooch crucible the ammonium chlorid solution is not in contact with the double salt longer than two or three minutes, errors from this source would not be expected to be and as our numerous results show are not appreciable.

There are few analytical methods where the precipitate is absolutely insoluble in the wash liquid, and it is absurd to discriminate against the Lindo-Gladding method for theoretical reasons which have no practicable significance.

It will be noticed that the results on kainit by direct evaporation with platinum solution are equally as good as when lime was previously separated. Calcium

¹ Land. Versuchs-Stat. 47, 97.

² Pogg. Ann., 29, 637; Zeitschr. f. Anal. Chem., 6, 216.

sulphate, when present in small quantities, is readily removed by ammonium chloride solution. We are indebted to De Roode¹ for calling attention to this fact; but, it should be remembered that *omission of previous separation of lime is merely returning to the plan first proposed by Gladding, from which many of us have never departed.*

FACTORS FOR THE CONVERSION OF POTASSIUM PLATINICHLORID TO POTASSIUM SULPHATE, CHLORID, AND OXID.

The factors which have been commonly used (0.3056 for converting K_2PtCl_6 to KCl , 0.3570 to K_2SO_4 and 0.19308 to K_2O), although based on old atomic weights, are those recommended by Fresenius (*Ztschr. Anal. Chem.*, **21**, 238), who found in some experiments performed shortly after Senbert's determination of the now accepted atomic weight of platinum that these factors gave results nearer theory than those derived from the revised atomic weights. It has, however, been shown (*J. Am. Chem. Soc.*, **17**, 453) that the method employed by Fresenius is at fault, yielding K_2PtCl_6 contaminated with included liquid, and for this reason the lower factors give the better results. The process adopted by this association at the last convention removes the above source of error, and the factors which are theoretically correct yield, practically, the best results. These are 0.1939 for conversion to K_2O , 0.3069 to KCl , and 0.3587 to K_2SO_4 .

If the figures given with last year's report on potash are examined, it will be noticed that these new factors correct a tendency toward low results.

All the results of the present year have also been calculated by the new factors. In order to save space the figures are not given in detail, but only the difference between K_2O found and K_2O taken, the average being used when two or more determinations were made by the same analyst. (See Table IV.) Where blanks were not made the results, which for this reason could not be corrected, are not taken into account. A comparison of the figures in Table IV will show that the new factors give better results than the old. The errors occasioned in the optional method by loss of potash through occlusion are not, of course, overcome, but the Lindo-Gladding method has been shown to be practically free from such errors and the results using the new factors are very close indeed to theory.

The mere fact that the Lindo-Gladding method gives the most accurate results by the use of the revised factors is of itself an indication of the perfection of the process and the purity of the double salt obtained. It is only in the case of methods which yield an impure double salt that there is need of resorting to the unscientific procedure of retaining incorrect factors in order to compensate for errors of the method.

It should be stated that the investigation of the year was planned as a test of methods, not of analysts. Since those taking part in it with hardly an exception proved themselves competent to execute careful analyses, it was possible to carry out the original purpose and as a result we have been able to arrive at important conclusions.

Too often in the past inexperienced chemists have made the analyses and their results when published have been used as evidence against our methods.

It should be the purpose of the association to study and perfect methods, and reporters should be relieved from serving as a sort of examining board for testing the qualifications of chemists.

Results of German chemists.—The following translation of Dr. Tietjen's report was made by Dr. von Herff, of the office of the German Kali Works, New York, and is therefore authoritative:

LEOPOLD SHALL, *Stassfurt, July 3, 1896.*

For the purpose of testing the merits of the Lindo-Gladding method and the Stassfurt method for the determination of potash, comparative analyses were made in several laboratories of the potash works and also in the laboratory of the syndicate. These investigations were made with samples furnished by the agricultural

experiment station at New Haven, Conn., and the results obtained again show that the Lindo-Gladding method, in comparison with the Stassfurt method, is troublesome and not accurate. Satisfactory results were obtained only in the analysis of kainit.

In the laboratory of the salt mine, New Stassfurt, the following results were obtained:

A.—*Muriate of potash.*

I. Pure muriate of potash according to the Stassfurt method.

1. From 0.2 g. substance were obtained 0.6526 g. 2KCl.PtCl_4 .
2. From 0.2 g. substance were obtained 0.653 g. 2KCl.PtCl_4 .
3. From 0.2 g. substance were obtained 0.653 g. 2KCl.PtCl_4 .

Average, 0.6528; 99.75 per cent KCl.

II. Muriate of potash 80 per cent according to the Stassfurt method.

1. From 0.2 g. muriate of potash = 0.419 g. 100 per cent KCl + .25 cc impurities were obtained 1.3745 2KCl.PtCl_4 ; calculated, 83.8 per cent; found, 84 per cent KCl.
2. From 0.423 g. muriate of potash = 0.422 g. 100 per cent KCl + .25 cc impurities were obtained 1.383 g. 2KCl.PtCl_4 ; calculated, 84.4 per cent; found, 84.53 per cent.
3. From 0.42 g. muriate of potash = 0.419 g. 100 per cent KCl + 25 cc impurities were obtained 1.368 g. 2KCl.PtCl_4 ; calculated, 83.8 per cent; found, 83.61 per cent.

Sample No. 3 was washed with alcohol, as prescribed in the Lindo-Gladding method, I.

III. Pure muriate of potash according to the Lindo-Gladding method, I.

1. From 0.5 g. muriate of potash were obtained 1.625 g. 2KCl.PtCl_4 = 99.32 per cent.
2. From 0.5 g. muriate of potash were obtained 1.6193 g. 2KCl.PtCl_4 = 99 per cent.
3. From 0.5 g. muriate of potash were obtained 1.621 g. 2KCl.PtCl_4 = 99.1 per cent.

IV. Muriate of potash 80 per cent according to the Lindo-Gladding method, I.

1. From 0.42 g. muriate of potash 0.419 g. 100 per cent KCl + 25 cc impurities were obtained 1.3665 g. 2KCl.PtCl_4 ; calculated KCl 83.8 per cent; found, 83.82 per cent.
2. From 0.421 g. muriate of potash 0.420 g. 100 per cent KCl + 25 cc impurities were obtained 1.3673 g. 2KCl.PtCl_4 ; calculated, 84.0 per cent; found, 83.57 per cent.
3. From 0.426 g. muriate of potash 0.425 g. 100 per cent KCl + 25 cc impurities were obtained 1.384 g. 2KCl.PtCl_4 ; calculated, 85.0 per cent; found, 84.59 per cent.

B.—*Sulphate of potash.*

I. Pure sulphate of potash according to the Stassfurt method.

1. From 0.2 g. sulphate of potash were obtained 0.5597 g. 2KCl.PtCl_4 = 99.7 + 0.3 = 100 per cent K_2SO_4 .
2. From 0.2 g. sulphate of potash were obtained 0.5604 g. 2KCl.PtCl_4 = 99.8 + 0.3 = 100.1 per cent K_2SO_4 .

II. Sulphate of potash 90 per cent according to the Stassfurt method. 1.84 g. of pure sulphate of potash + 100 cc impurities at 200 cc, of which 20 cc were evaporated.

1. From 0.2 g. substance were obtained 0.5129 g. 2KCl.PtCl_4 ; calculated, 92 per cent, K_2SO_4 ; found, 91.45 + 0.3 = 91.75 per cent.
2. From 0.2 g. substance were obtained 0.5134 g. 2KCl.PtCl_4 ; found, 91.54 + 0.3 = 91.84 per cent.

III. Pure sulphate of potash according to the Lindo-Gladding method, I.

1. From 0.5 g. sulphate of potash were obtained 1.4191 g. 2KCl.PtCl_4 = 101.2 per cent K_2SO_4 .
2. From 0.5 g. sulphate of potash were obtained 1.4188 g. 2KCl.PtCl_4 = 101.18 per cent K_2SO_4 .
3. From 0.5 g. sulphate of potash were obtained 1.407 g. 2KCl.PtCl_4 = 100.35 per cent K_2SO_4 .

IV. Sulphate of potash 90 per cent according to Lindo-Gladding method, I.

1. From 0.461 g. sulphate of potash + 25 cc impurities were obtained 1.3056 g. 2KCl.PtCl_4 ; calculated K_2SO_4 , 92.2 per cent; found, 93.11 per cent.
2. From 0.46 g. sulphate of potash + 25 cc impurities were obtained 1.2921 g. 2KCl.PtCl_4 ; calculated, K_2SO_4 , 92.4 per cent; found, 92.15 per cent.
3. From 0.46 g. sulphate of potash + 25 cc impurities were obtained 1.3035 g. 2KCl.PtCl_4 ; calculated, K_2SO_4 , 92 per cent; found, 92.96 per cent.

V. Pure sulphate of potash according to Lindo-Gladding method, II.

1. From 0.5 g. sulphate of potash were obtained 1.360 g. 2KCl.PtCl_4 = 97 per cent K_2SO_4 .
2. From 0.5 g. sulphate of potash were obtained 1.3784 g. 2KCl.PtCl_4 = 98.3 per cent K_2SO_4 .
3. From 0.5 g. sulphate of potash were obtained 1.3775 g. 2KCl.PtCl_4 = 98.2 per cent K_2SO_4 .
4. From 0.5 g. sulphate of potash were obtained 1.369 g. 2KCl.PtCl_4 = 97.64 per cent K_2SO_4 .
5. From 0.5 g. sulphate of potash were obtained 1.374 g. 2KCl.PtCl_4 = 98 per cent K_2SO_4 .

C.—*Sulphate of potash-magnesia.*

- I. According to the Stassfurt method, 1.04 g. pure sulphate of potash + 100 cc impurities at 200 cc, of which 20 cc were evaporated.
1. From 0.2 g. substance were obtained 0.2916 g. 2KCIPtCl_4 ; calculated, K_2SO_4 , 52 per cent; found, 51.99 per cent.
 2. From 0.2 g. substance were obtained 0.2908 g. 2KCIPtCl_4 ; found, 51.85 per cent.
- II. According to Lindo-Gladding method, I.
1. From 0.262 g. sulphate of potash + 25 cc impurities were obtained 0.7507 g. 2KCIPtCl_4 ; calculated, 52.4 per cent; found, 53.54 per cent.
 2. From 0.260 g. sulphate of potash + 25 cc impurities were obtained 0.7372 g. 2KCIPtCl_4 ; calculated, 52 per cent; found, 52.58 per cent.
 3. From 0.261 g. sulphate of potash + 25 cc impurities were obtained 0.7322 g. 2KCIPtCl_4 ; calculated, 52.2 per cent; found, 52.2 per cent.

D.—*Kainit.*

- I. According to the Stassfurt method, 0.7 g. pure sulphate of potash + 125 cc impurities at 250, of which 20 cc were evaporated.
1. From 0.2 g. substance were obtained 0.1565 g. 2KCIPtCl_4 ; calculated, K_2SO_4 , 28 per cent; found, 27.9 per cent.
 2. From 0.2 g. substance were obtained 0.1573 g. 2KCIPtCl_4 ; found, 28.1 per cent.
- II. According to the Lindo-Gladding method, I, without removal of lime.
1. From 0.1444 g. sulphate of potash + 25 cc impurities were obtained 0.4084 g. 2KCIPtCl_4 ; calculated, 28.88 per cent; found, 29.16 per cent K_2SO_4 .
 2. From 0.140 g. sulphate of potash + 25 cc impurities were obtained 0.3908 g. 2KCIPtCl_4 ; calculated, 28 per cent; found, 27.9 per cent K_2SO_4 .
 3. From 0.140 g. sulphate of potash + 25 cc impurities were obtained 0.3960 g. 2KCIPtCl_4 ; calculated, 28 per cent; found, 28.25 per cent K_2SO_4 .

The results reported by the Consolidated Alkali Works at Westeregeln have reference only to kainit. An artificial kainit was produced by means of pure substances furnished by Mr. Winton. This contained in 50 g. 12.540 g. K_2SO_4 , therefore 25.08 per cent.

In the analysis of this kainit the following results were obtained by the Stassfurt method: From 0.4 g. substance were obtained 0.2810 K_2PtCl_6 = 25.08 per cent K_2SO_4 .

With the Lindo-Gladding method the following results were obtained: (a) With preceding precipitation of lime, from 0.5 g. substance were obtained 0.3535, 0.3575 K_2PtCl_6 = 25.22 per cent, 25.52 per cent K_2SO_4 . (b) Lime not precipitated, from 0.5 g. substance were obtained 0.3575 K_2PtCl_6 = 25.52 per cent K_2SO_4 .

From the laboratory of the syndicate the following results were obtained:

- A. 0.3056 pure muriate of potash, free from water, mixed with impurities corresponding to the 80 per cent muriate of potash.
1. According to the Stassfurt method, 1.000, 1.001, 1.000 K_2PtCl_6 .
 2. According to the Lindo-Gladding method, 0.991, 0.993 K_2PtCl_6 .
- B. 0.357 pure sulphate of potash, free from water, mixed with impurities corresponding to the 90 per cent sulphate of potash.
1. According to the Stassfurt method, 0.995 g., 0.995, 0.996 K_2PtCl_6 .
 2. According to the Lindo-Gladding method, 1.013 g., 1.012 K_2PtCl_6 .
- C. 0.2 g. pure sulphate of potash, mixed with impurities corresponding to the potash-magnesia.
1. According to Stassfurt method, 0.557 K_2PtCl_6 , 0.1988 K_2SO_4 , 0.559 K_2PtCl_6 = 0.1996 K_2SO_4 .
 2. According to Lindo-Gladding method, 0.5686 K_2PtCl_6 = 0.2030 K_2SO_4 .
- D. 0.2 g. pure sulphate of potash, mixed with impurities corresponding to the kainit.
1. According to the Stassfurt method, 0.560, 0.561 K_2PtCl_6 = 0.200, 0.2003 K_2SO_4 .
 2. According to the Lindo-Gladding method, 0.562, 0.561 K_2PtCl_6 = 0.2006, 0.2003 K_2SO_4 .

DR. TIETJENS,

For the Sworn Chemists of the Sales-Syndicate of the Potash Works.

TABLE V.—Average results from German chemists connected with the Stassfurt industry.

[Old factors.]

	Stassfurt method.				Lindo-Gladding method.			
	KCl or K ₂ SO ₄ taken.	KCl or K ₂ SO ₄ found.	KCl or K ₂ SO ₄ error.	K ₂ O error.	KCl or K ₂ SO ₄ taken.	KCl or K ₂ SO ₄ found.	KCl or K ₂ SO ₄ error.	K ₂ O error.
Potassium chlorid, c. p.:	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>
Neu-Stassfurt	0.1991	0.1995	+0.0004	+0.0002	0.4977	0.4956	—0.0021	—0.0013
Syndicate Laboratory3056	.3057	+ .0001	+ .0001	.3056	.3032	— .0024	— .0015
Muriate of potash, 80 per cent:								
Neu-Stassfurt4191	.4202	+ .0011	+ .0006	.4204	.4195	— .0009	— .0005
Potassium sulphate, c. p.:								
Neu-Stassfurt2000	.2005	+ .0005	+ .0003	.5000	.5052	+ .0052	+ .0028
Sulphate of potash, 90 per cent:								
Neu-Stassfurt1840	.1838	— .0002	— .0001	.4603	.4642	+ .0039	+ .0021
Syndicate Laboratory3570	.3564	— .0006	— .0003	.3570	.3615	+ .0045	+ .0024
Sulphate of potash and magnesia:								
Neu-Stassfurt1040	.1039	— .0001	— .0001	.2610	.2642	+ .0032	+ .0017
Syndicate Laboratory2000	.1992	— .0008	— .0004	.2000	.2030	+ .0030	+ .0016
Kainit:								
Neu Stassfurt0560	.0560	.0000	.0000	¹ .1415	.1422	+ .0007	+ .0004
Westeregeln1003	.1003	.0000	.0000	² .1254	.1269	+ .0015	+ .0008
Syndicate Laboratory2000	.2001	+ .0001	+ .0001	¹ .1254	.1276	+ .0022	+ .0014
Potassium sulphate, c. p., op-					² .2000	.2004	+ .0004	+ .0002
tional method:								
Neu-Stassfurt5000	.4897	— .0103	— .0056

¹ Lime not precipitated.² Lime precipitated.

In Table V are given the average figures obtained in each of the three laboratories, calculated by the old factors, from which it appears that while the German chemists obtained very accurate results by their own method, they had, as a rule, poor success with ours.

The Lindo-Gladding method gave low results on potassium chlorid, with and without addition of impurities, while on the sulphate containing salts they were, as a rule, very much too high.

At Neu-Stassfurt seven determinations were made by the optional method on pure potassium sulphate, with an average loss of 0.0103 gram.

In view of the accurate results which were obtained by the Lindo-Gladding method in eight American laboratories, it seems probable that the German chemists carried out the details of the process differently. Possibly they did not use the Gooch crucible, which in our laboratories is identified with the method.

AMENDED AND ADOPTED REPORT OF THE COMMITTEE OF RECOMMENDATIONS IN RELATION TO POTASH.

(1) That the following be used as factors in computing potash results instead of those now in use: Factors for KCl=.3069; factors for K₂SO₄=.3587; factors for K₂O=.1939.

(2) That a careful test of the accuracy of the Stassfurt method be made by the next reporter by the use of pure potash salt in conjunction with impurities corresponding to those found in the potash salts.

(3) That the next reporter have a careful test made of the accuracy of the optional method, by acidulating the solution before precipitating the sulphuric acid.

(4) That the following resolution be adopted: *Resolved*, That it is the sense of this

association that in mixed fertilizers chlorin should be taken into consideration in reporting upon their composition.

(5) That the reporter for 1897 be requested to test the accuracy of kainit determinations by the Lindo-Gladding method without previous removal of lime and sulphuric acid.

(6) That page 22 (2) (b) be changed to read as follows: When it is desired to determine the total amount of potash in organic substances, like cotton-seed meal, etc.

(7) That Mr. Winton be authorized to revise the phraseology of the description of the potash methods.

H. J. WHEELER,

For Committee on Recommendations of Reporters.

Convention adjourned until 1.30 p. m.

FRIDAY—AFTERNOON SESSION.

The president called for papers on the subject of the determination of potash in fertilizers.

Mr. CURTIS. I desire to present a paper by Mr. Scovell, Mr. Peter, and myself, entitled "Some sources of error in the determination of potash in fertilizers."

The PRESIDENT. Before the presentation of the paper we will hear the report of the committee appointed to invite the Secretary and Assistant Secretary of Agriculture to attend our meetings.

Messrs. Frear and Lindsey, the committee appointed to wait upon the Secretary of Agriculture, reported that the Secretary was unable to attend, owing to his being present at the Cabinet meeting. The Assistant Secretary has promised to be present at some time during the meetings of the association.

ON SOME SOURCES OF ERROR IN THE DETERMINATION OF POTASH IN FERTILIZERS.

By M. A. SCOVELL, A. M. PETER, and H. E. CURTIS.

The investigation described in this paper was occasioned by the appearance of certain small but annoying and seemingly unaccountable discrepancies, in the case of certain fertilizers, between the potash determinations made by two of the chemists of this laboratory when working on the same samples and with the same apparatus and reagents. For instance, in a fertilizer composed of acid phosphate, tankage, dried blood, and muriate of potash (No. 3345), one chemist obtained 1.68 and 1.71 per cent potash, while the other obtained 1.80 and 1.80 per cent; in another similar fertilizer (No. 3346) one obtained 1.76 and 1.83, while the other obtained 1.92 and 1.96. Upon carefully comparing their manipulation in carrying out the official Lindo-Gladding method, the only difference found was that the one who usually obtained the lowest results, always measured the 300 cc of water with which the substance was to be boiled, while the other was in the habit of pouring upon the substance as much water as he judged to be not less than 300 cc, and probably exceeded that amount in nearly every case. The further observation that the differences did not appear when both chemists worked on the same solution, but only when each prepared a separate solution of the fertilizer for analysis confirmed the belief that the cause was to be sought in some difference of manipulation in preparing the solution. One of us (Curtis) suggested the theory that the spongy material, such as tankage, etc., in the fertilizer, having become saturated with the solution

containing potash during the boiling, retains this solution in its original concentration when the liquid is made up to volume and filtered. If, then, the fertilizer be boiled with, say, 400 cc of water, the solution retained will be weaker and the final result will be higher than where only 300 cc were used, assuming evaporation to be about the same in each case. On the other hand, supposing the same quantity of water taken in each case, if the boiling be brisk and the evaporation rapid the solution will be more concentrated than if it were slow; the solution retained will be stronger and the final result lower. The following results seem to point to the correctness of this theory:

Method.	3346.	3040.	3042.	3347.	3404.	3407.	Tankage mixture.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
K ₂ O. L. G. method, boiling with 300 cc water.	1.85	2.51	1.66	2.48	1.63	2.34	4.95
Same, boiling with 400 to 450 cc water.	1.82
	1.93	2.57	1.71	2.71	1.61	2.42	5.02
	1.97
Gain115	.06	.05	.25	.02	.08	.07

In the series where 300 cc of water were used the boiling was purposely brisk and the evaporation amounted 40 or 50 cc in each case; in the other series the boiling was slow and evaporation very little. In the first series the residue at the end of the boiling would be saturated with a solution containing the potash in about 250 cc of liquid, while in the second the same amount of potash would be contained in more than 400 cc. The fertilizer 3346 was composed of acid phosphate, tankage, dried blood, and muriate of potash; 3040 and 3042 contained acid phosphate and cotton-seed meal; 3347 was an acid phosphate containing no organic matter with muriate of potash; 3404 contained tankage and dried blood, and only showed 3 per cent water soluble phosphoric acid; 3407 was an acid phosphate with potash, but no organic material, and the last was a mixture of tankage, acid phosphate, and muriate of potash. It will be noted that the difference is greater in the case of the plain acid phosphates than in the others, where organic materials were present.

To further test the theory of occlusion by porous materials some experiments were made by pouring a solution of potash salt of known strength upon filter paper or chamois skin in a 500 cc flask, and after standing a short time, making up to volume, filtering, and determining the potash in 500 cc of the filtrate, as in the regular course of analysis. The results are stated as platinum salt obtained, and also as per cent of potash, calculated as if obtained from 1 gram of fertilizer.

The potash solution used contained 0.05 gram c. p. KCl in 50 cc.

Materials and time.	K ₂ PtCl ₆ obtained (gram).	Per cent K ₂ O.
50 cc KCl solution alone	{ 0.1608 .1612 }	3.11
50 cc KCl and 10 grams filter paper. After 1 hour it was made up to volume, shaken well, and filtered.	{ .1366 .1353 }	2.62
50 cc KCl and 6 grams chamois skin, cut fine, instead of paper	{ .1403 .1399 }	2.71
50 cc KCl and 250 cc water with 5 grams filter paper. Stood one-half hour before making up to volume.....	.1598	3.08
50 cc KCl in same way, but stood 36 hours after making up to volume before filtering.....	.1599	3.08
50 cc KCl in same way as the next preceding, but used 9 grams paper1588	2.90

The effect of occlusion of the solution in the porous material appears very distinctly in the first two experiments in which KCl was used in less dilute solution than in the others.

To determine whether a precipitate of phosphates acts in the same way, 10 grams of precipitated tricalcium phosphate were boiled one-half hour with 10 cc dilute sulphuric acid, a known quantity of the KCl solution, and enough water to make about 300 cc. Ammonia and ammonium oxalate were then added, the volume was made up to 503.5 cc, allowing $3\frac{1}{2}$ cc for volume of precipitate, and the determination finished as in the regular analysis. The results are stated as platinum salt obtained from 50 cc of filtrate and also as percentage of K_2O calculated as from 1 gram. The weight of platinum salt has been diminished by 0.0009, the amount obtained in a blank experiment with the precipitated phosphate.

Quantities used.	K_2PtCl_6 (gram).	Per cent K_2O .
50 cc KCl solution, alone (from previous experiment).....	0.1608	3.11
	.1617	
50 cc KCl, 10 grams tricalcium phosphate, etc., stood over night after adding the ammonia and ammonium oxalate and before making up to volume.	.1544	2.96
	.1525	
Deficit.....	.0078	.15
100 cc KCl solution, alone (from previous experiment).....	.3225	6.22
100 cc KCl, 10 grams tricalcic phosphate as before.....	.3118	6.02
	.3124	
Deficit.....	.0104	.20

It thus appears that the precipitated phosphates retain a notable amount of potash, and this in a more marked degree than the organic materials experimented upon. The foregoing results seem to indicate that the absolute error in analysis due to retention of potash by the phosphates is quite noteworthy. It is true that in making up the solution to 500 cc, without allowing for the volume occupied by the fertilizer, we introduce an error in the opposite direction, but as 10 grams of fertilizer do not occupy more than about 4 cc it is evident that this compensation may not always be sufficient. We see also in this source of error a reason why the "alternate" method should give slightly higher results, as a rule, than the Lindo-Gladding.

POTASSIUM (MONO) METAPHOSPHATE.

In the experiments with tricalcium phosphate just described the first series of determinations made gave a very remarkable result, the figures obtained being 2.57, 1.35, 0.50, 1.78, 2.77, and 1.94 per cent KO_2 , the theoretical amount being 3.11 per cent. It was noticed that the porcelain dishes in which the residues had been ignited for the expulsion of ammonia salts were much corroded, and on adding water with a few drops of hydrochloric acid to the ignited residue it was found that a quantity of white substance in the form of glassy scales remained undissolved. This was supposed to be silica and was filtered out before adding platinic chlorid. As soon as it appeared that there had been a serious loss of potash somewhere, this white substance was examined more carefully and was found to contain the insoluble modification of potassium metaphosphate (mono-metaphosphate) as well as silica, etc., from the porcelain. The solution for analysis prepared as described with tricalcic phosphate contained ammonium phosphate. On evaporation and ignition there remained an excess of phosphoric acid, and the potash was, of course, converted into (mono) metaphosphate. This substance is almost insoluble in pure water, but dissolves in a short time when digested on the water bath with water acidified with hydrochloric acid. We committed the error of assuming that because nothing but a potash salt was present it would dissolve readily in hot water and did not allow time enough for the solution of the metaphosphate in the dilute acid.

Of course the filters were thoroughly washed with hot water, but this simply removed the acid liquid without affecting the solution of the metaphosphate.

Potassium (mono) metaphosphate was prepared and analyzed by Maddrell (Liebig's *Annalen*, LXI, p. 53), and is formed when potassium chlorid or other potash salt is heated with excess of phosphoric acid. We prepared some of the pure salt and found it to be almost insoluble in cold water and very slowly soluble in boiling water with conversion into orthophosphate. An experiment with cold water gave its solubility as about 1 part in 5,000. The solubility in boiling water depended on the length of time of boiling. The finely powdered salt dissolved easily in warm dilute acids, even in acetic acid. It appeared to be not sensibly volatile on ignition to a bright red heat, but lost weight on ignition with a blast lamp.

The frequent presence of soluble phosphates in the solutions of fertilizers prepared by the Lindo-Gladding method has been pointed out by Huston and a remedy suggested. Their presence is objectionable on account of the action of the phosphoric acid on the dishes in which the residue is ignited. If these dishes are of porcelain, a quantity of finely divided silica is frequently obtained in the platinum precipitate, rendering it slow of filtration. Besides this it appears from our experience that a loss of potash is sometimes possible if sufficient attention is not paid to dissolving the ignited residue with water and hydrochloric acid. In the analysis of fertilizers it frequently happens that there is a slight residue which will not dissolve even after protracted digestion, but we should always allow time enough to insure complete solution of the potash compounds.

KENTUCKY AGRICULTURAL EXPERIMENT STATION,

Lexington, Ky., November 6, 1896.

After the reading of the recommendations the president called for a discussion of the paper.

Mr. Wiley moved that on account of the importance of bringing American and European methods for estimating potash into harmony and for the purpose of pointing out some mistakes in the attacks which have been made on the method followed in this country, Messrs. Winton and Wheeler be requested to prepare a paper on the official Lindo-Gladding method for estimating potash for publication in the *Versuchsstationen*.

THE PRESIDENT. I think it is very appropriate to take some such action as this.

The motion was adopted.

Mr. SHUTT. I should like to inquire if the determination of potash in soils should properly be exhibited now or come up later, when the report on soils is read.

THE PRESIDENT. It is better, I think, to discuss that subject when the report on soil analysis is under consideration. If there is no further discussion of the subject, the recommendations of the reporter on potash will be referred to the committee, in harmony with the usual order. Perhaps we had better take up now the next subject, viz, phosphoric acid.

Mr. McDONNELL. I would like to have the report on phosphoric acid deferred until to-morrow.

It was voted that the phosphoric acid report be deferred, and that the report on soils take its place.

Mr. PATTERSON. It seems to me, Mr. President, that we should not defer reports because of the absence of certain members of the

association who desire to hear them. The order of business should be arranged for the benefit of those who are present, and not for those who are absent.

Mr. GOSS. It was not expected, Mr. President, that the report on soils should come up at the present time, and hence it is not fair to those who are absent to have it brought forward now, when the regular order of business calls for something else.

The PRESIDENT. It seems advisable in the circumstances to let the report on soils go over. I would like to ask if Mr. Lindsey is ready to report on foods and feeding stuffs?

Mr. LINDSEY. I think now is perhaps as good a time as any to present my report, as I suppose there are very few in the association who are interested in it any way.

The PRESIDENT. Mr. Lindsey will then present his report on foods and feeding stuffs.

REPORT ON ANALYSIS OF CATTLE FEEDS.

By J. B. LINDSEY.

Your reporter for the present year has not considered it wise to attempt any further comparison of results obtained by different analysts in determinations of moisture, crude ash, fiber, fat, and protein. He believes these methods to be as perfect as they ever can be made, and when carried out by experienced chemists will give at least comparative results. It is generally recognized that these terms do not refer to single substances, but rather to groups of ingredients possessing similar characteristics.

For example, investigation has shown that crude fiber prepared from coarse fodders, brans, and the like, usually consists principally of dextroso-cellulose, combined with the small quantities of lignin or lignin acids not removed by the action of dilute acid and alkali. It also contains more or less pentosans, so closely identified with the dextroso-cellulose and lignin, as to be considered a pentoso-cellulose. If one, therefore, desires to continue investigations along this line, the aim should be to secure a method for the determination of cellulose separated from other ingredients. That this is at present surrounded with difficulty is clear, from the fact that as yet we have not a very exact knowledge of the several modifications of cellulose.

Again, it is known that ether extract comprises not only the true fats, but also the resins, waxes, coloring matter, cholesterin, and lecithin. If one would study this class of substances, the aim should be to perfect methods to estimate them separately. No single method, no matter how much we may compare individual results, will do this.

The admirable work of Osborne at the Connecticut Experiment Station is throwing much additional light upon the meaning of crude protein. We must be content with the collective estimation of albuminoids and nonalbuminoid nitrogen-containing bodies until investigators have thoroughly studied the many different forms of protein that exist in plants and seeds.

The term "nitrogen-free extract" has had until recently a very doubtful meaning. It has been said to consist of starch, sugar, gums, etc. It included everything known and unknown that could not be classified elsewhere. Thanks to the investigation of Tollens,¹ Schulze,² and their pupils, a great deal of light has been thrown upon this hitherto questionable combination. In addition to starch and the small quantities of ready formed sugars of the plant, it has been found to contain the larger

¹ Landw. Vers. Sta., 39, p. 401, and Journal für Landw., 1896.

² Zeitsch für Physiol. Chem. Bds., 14-16.

part of the lignin, removed from combination with the cellulose by the action of the acid and alkali, and a group of bodies called hemicelluloses, which are distinguished from the true cellulose by their solubility in dilute mineral acids and in F. Schulze's reagent, and by their insolubility in Schweizer's reagent. The hemicelluloses include xylan and araban or pentosans, dextran laevulan, mannan and galactan, which, when hydrolyzed with dilute acid, yield the sugars xylose, arabinose, dextrose, laevulose, mannose, and galactose. Investigations have shown the pentosans to be by far the most generally distributed, followed next by galactan.

From these few remarks it will be seen that future work on cattle feeds ought to a considerable extent to be in the line of direct investigation. One must identify and study the nature of the substances composing plants before attempting to estimate them quantitatively.

Your reporter for this year felt that it would not be wise to undertake too great a variety of work, but rather to concentrate upon one or two special lines. It seemed wise to continue the work marked out by Mr. Patterson a year ago and test methods that have been proposed for the estimation of individual substances comprising the extract matter. Invitations were accordingly sent out early in November, 1895, to as many as it seemed would be interested in the work. Fifteen replies were received, to which samples and instructions were duly sent.

Three of this number have failed to send results or any excuse. Seven during the month of October have stated their inability from one cause and another to carry out the work, so that we have actual results from but six chemists, including our own station.

The lines of work proposed were as follows:

A. *Estimation of starch by three distinct methods:*

I. Sachsse's method.

II. Diastase method.

III. Mäcker's method.

B. *Estimation of pentosans using method perfected by Mann, Flint, and Tollens.*

C. *Estimation of galactan by method of Rischbieth, Credyt, Hadecke, and Tollens.*

A.—STARCH.

Two samples were sent out—(I) potatoes and (II) Buffalo gluten feed. This latter consists of hull, germ, and gluten of Indian corn. The samples were both finely ground so as to pass through a 12-mm. mesh sieve.

I.—SACHSSE'S METHOD.

Extract 4 to 5 grams of the substance with ether, then treat the residue in a beaker with 50 cc of 10 per cent alcohol for half an hour with constant stirring, filter and wash with strong alcohol. Bring the residue with 200 cc of water and 20 cc of 25 per cent hydrochloric acid (sp. gr. 1.125) into a 10-ounce flask provided with a reflux condenser and heat in a boiling-water bath for three hours, cool and very nearly neutralize with sodium carbonate (avoiding excess). Clarify if necessary with 10 cc basic acetate of lead and make up to 500 cc. Pour off through a dry filter and determine the dextrose in an aliquot part by Allihn's method. Convert the weight of the dextrose to that of starch by the factor 0.9.

II.—DIASTASE METHOD.

Free 3 to 5 grams of the substance from fat and water soluble carbohydrates, as under Sachsse's method. Bring the residue into a beaker with 50 cc of water, and immerse in a boiling-water bath, stirring constantly until the starch forms a paste. Cool to 65° C. and add 25 cc of malt extract, and maintain at this temperature until the solution no longer gives the starch reaction with iodine. Filter into a 10-ounce flask and wash until the filtrate amounts to 200 cc. Then add 20 cc of 25 per cent hydrochloric acid (sp. gr. 1.125), connect with a reflux condenser, and proceed as under Sachsse's method.

III.—MÄRCKER'S METHOD.

Extract from 3 to 5 grams of the substance with ether to remove fat and then bring the substance into a beaker with 50 cc of 10 per cent alcohol and allow to stand for half an hour with frequent stirring. Filter, wash with alcohol, and allow to dry. Remove dry substance from filter, make fine, and bring into a Linter pressure bottle (if Soxhlet digester be not at hand), add 50 cc of water, and immerse bottle in boiling water till starch is all brought into a jelly (stirring frequently). Cool to 65° C., add 10 cc malt extract, and digest at 65° C. for one hour. Now add 5 cc of a 1 per cent tartaric acid solution and heat at 140° C. for one-half hour, or 110° C. for one hour. Cool to 65° C., add 10 cc malt extract, and maintain at this temperature until solution no longer gives starch reaction with iodine; then filter into a 10-ounce flask, wash until the filtrate measures 200 cc., add 20 cc HCl of 1.125 sp. gr., and heat the flask, to which is fitted a reflux condenser, in a boiling-water bath for two and one-half hours. Neutralize with Na_2CO_3 , filter if necessary, and make up to 500 cc. Determine dextrose as in Allihn's method.

Prepare the malt extract by digesting 10 grams of fresh, finely ground malt over night, at ordinary room temperature, with about 175 cc of water, filter, and make to 200 cc. Determine the amount of dextrose in a given quantity by treating it the same as with the determination of starch.

Starch determinations.

	I. Potatoes.			II. Buffalo gluten feed.		
	Sachsse's method.	Diastase method.	Märcker's method.	Sachsse's method.	Diastase method.	Märcker's method.
United States Department of Agriculture, W. H. Krug	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Wisconsin Experiment Station F. W. Woll.....	68.23	62.69	39.91	21.90	19.84
Vermont Experiment Station, C. H. Jones	68.40	63.32	61.90	38.53	21.54	21.00
New Hampshire Experiment Station, C. D. Howard	70.41	64.19	38.70	21.14
Columbia University, H. C. Sherman..	77.48	67.15	68.00	42.37	22.71	23.36
Massachusetts Experiment Station, E. B. Holland.....	69.80	66.73	69.15	38.60	20.85	25.27
Average	69.21	64.82	68.57	38.93	21.29	22.37

The results are so few that one can draw no very accurate conclusions. On potatoes, the figures obtained by Woll, Jones, Howard, and Holland with Sachsse's method agree quite well and represent the amount of starch usually found. Sherman's results are without doubt too high. With the diastase method on the same substance, Sherman and Holland obtain practically identical results. In case of Märcker's method, Sherman and Holland again agree, and both seem to have secured higher results than with the diastase method. With the potatoes, Sachsse's method appears to give slightly higher results than either of the others.

Coming to the estimation of starch in the gluten feed, marked differences are noted. Sachsse's method shows some variations, but not extreme ones. For some reason Sherman secures a higher amount of starch by this method in case of both samples. Both the diastase and Märcker methods agree fairly well, but show very much lower results than those obtained by Sachsse's method. This is to be expected, for the gluten feed contains 16 or more per cent of pentosans, which substance, as well as all hemi-celluloses, is converted into sugars by the action of dilute acids. The fact is firmly established that Sachsse's method is entirely unreliable for the

determination of starch in all cases where any of the hemi-celluloses are present. Recent investigations limit its value.

Märcker's method as a rule gives results slightly higher than the diastase method. This may be possibly due to the action of pressure on carbohydrate bodies other than starch.

I desire in this connection to present the results of Mr. H. C. Sherman, recently published.¹

	Sachsse's method.	Diastase method.	Märcker's method.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Pure starch	82.30	82.49	82.39
Wheat flour	68.35	66.55	67.15
Oatmeal	59.01	56.23	56.16
Graham flour	58.63	55.32
Wheat bran	38.82	20.97	21.09
Wheat straw	22.69	4.39	4.80
Corn fodder	20.13	.96

The above figures confirm the results previously given.

OBJECTIONS TO THE DIASTASE AND MÄRCKER METHODS.

First. We are not positive that the diastase has no solvent action upon the hemi-celluloses. Stone's work would indicate, however, that the pentosans are not attacked, and it is probable that the action, if any, is slight.

Second. The inconvenience of being obliged to prepare frequent malt solutions, and of estimating the amount of reducing sugar they contain.

Third. The additional inconvenience, should Märcker's method be employed, of heating under pressure.

It is to be hoped that some method will be devised in the near future that will do away with these difficulties.

Your reporter feels justified in recommending as provisional methods for the estimation of starch the so-called diastase and Märcker methods. The Sachsse's method is entirely out of the question.

If conveniences are at hand for working the Märcker method, it should have the preference.

B. ESTIMATION OF PENTOSANS.

In view of the very general occurrence of the pentosans in plants and seeds, it seemed advisable to familiarize chemists with the method employed in their quantitative estimation and to see if closely agreeing results could be obtained with the method in the hands of different analysts.

The method is the one worked out by Flint, De Chalmot, and Tollens and is described as follows:

Weigh out 3 to 5 grams (depending on the amount of pentosans present) of the finely ground material, and transfer to a 300 cc flask. After adding 100 cc of hydrochloric acid of 1.06 sp. gr., connect with a Liebig condenser, and distil 30 cc, taking ten to fifteen minutes for the distillation. Instead of putting the flask in Rose's metal to diffuse the heat, as Flint and others suggest, we have placed it upon a piece of gauze with equally satisfactory results. Now add 30 cc more acid by means of a separatory funnel (the stem of which passes through the cork into the flask), and so continue the process until a drop of the distillate gives no red coloration on filter paper that has been moistened with anilin acetate (a few drops of anilin in a little 50 per cent acetic acid). Ten to eleven distillations are generally sufficient. Flint has shown that for constant results the presence of a certain amount of salt in a definite quantity of solution is essential. Furfurol hydrazone is

¹ School of Mines Quarterly, Vol. XVII, No. 4.

more insoluble with 81 grams of sodium chlorid in 400 cc of distillate than when less is present, and these proportions should be always used. Add to the obtained distillate, brought into a 700 to 800 cc beaker, the necessary amount of salt and water to raise it to this standard (see table following).

Distillate (cubic centi- meters).	Water (cubic centimeters).	Salt to be add- ed (grams).	Distillate (cubic centi- meters).	Water (cubic centimeters).	Salt to be add- ed (grams).
400	-----	-----	150	250	50.75
350	50	10.15	100	300	60.90
300	100	20.30	50	350	71.05
250	150	30.45	-----	400	81.20
200	200	40.60			

Neutralize exactly the 400 cc solution with sodium carbonate, covering the beaker with a watch glass during the process, and make up to 500 cc with water. Add now 10 cc of phenylhydrazin solution,¹ precipitating the furfural as furfural hydrazone.² Stir thirty minutes, preferably with an automatic stirrer, and filter the solution, using suction, into glass drying tubes about three-fourths inch in diameter and 6 to 7 inches long, drawn out at the lower end and filled with about one-half inch of glass wool in preference to asbestos. Remove adhering particles with a feather, and do not use over 100 cc of wash water. Dry the precipitate in a specially constructed air bath³ at 55° to 60° C. for three hours. To hasten and perfect the drying a partial vacuum is made by drawing dry air slowly through the tubes by aid of a suction pump, the air supply being regulated by pinchcocks. The air before entering the tubes is conducted through sulphuric acid to dry it, and through a glass tube containing small pieces of marble to remove any sulphuric acid that might be carried over mechanically. Cool the tubes in a desiccator and weigh. Dissolve out the precipitate with hot alcohol and reweigh, and consider the loss in weight as furfural hydrazone.

For the conversion of furfural hydrazone to furfural use the following factors:⁴

Furfural = furfural hydrazone \times 0.538.

Arabinose = furfural hydrazone \times 1.229 + 0.0177.

Xylose = furfural hydrazone \times 1.031 - 0.001.

Pentose (average arabinose and xylose) = furfural hydrazone \times 1.13 + 0.0083.

Pentosan = pentose \times 0.88.

Formulas.

Furfural = C_4H_3O-COH .

Phenyl-hydrazin = $C_6H_5-NH-NH_2$.

Furfural hydrazone = $C_4H_3OCHC_6H_5N_2H$.

Pentose = $(C_5H_{10}O_5)_n$.

Pentosans = $(C_5H_8O_4)_n$.

determination of pentosans.

Analysts.	Sample II, Buffalo glu- ten feed.	Sample IV, blue lupine.
	<i>Per cent.</i>	<i>Per cent.</i>
United States Department of Agriculture, William H. Krug	19.80	17.45
New Hampshire Station, C. D. Howard	16.39	9.94
Columbia University, H. C. Sherman	15.30	8.75
Massachusetts Station:		
J. B. Lindsey	-----	-----
E. B. Holland	16.77	9.39
Average	16.15	9.36

a Excluded from average.

¹Twelve grams of phenylhydrazin and 7.5 grams of glacial acetic acid filled to 100 cc with water and well shaken.

²After the addition of the phenylhydrazin reagent, the solution should react slightly acid. In case this is not so, add the necessary acetic acid.

³See Twelfth Report of Mass. State Exp. Sta. 1894.

⁴See factors recommended further on, to be used in the future.

The method requires considerable time in its execution, and some practice is necessary in order to secure good results. The above figures therefore are quite encouraging. We note the presence of about 16 per cent of pentosans in the gluten feed, and nearly 9 (or one-half) per cent in the lupine.

RECOMMENDATION.

The place of the factors given on page 63 of the Methods of Analysis of the A. O. A. C. for 1895, for the conversion of the hydrazone into pentosans or of those in the preceding pages, I would recommend the adoption of the following:

Weight of hydrazone $\times 0.516 + 0.0104 \times 1.81 \div$ weight of substance taken = pentosans.

Pentosans $\div 88 =$ pentoses.

Hydrazone $\times 0.516 + 0.0104 \times 1.65 \div$ amount substance taken = xylan.

Hydrazone $\times 0.516 + 0.0104 \times 2.03 \div$ amount substance taken = araban.

Xylan or araban $\div 0.88 =$ xylose or arabinose.

These factors have been very carefully worked out by Mann, Kruger, and Tollens.¹ It is well known that the pentosans, when distilled with hydrochloric acid, are somewhat decomposed. It is therefore not possible to calculate from the theoretical formula of the furfural hydrazone, the amount of pentosans actually present in the substance. Tollens has distilled known amounts of pure furfural, xylose, or arabinose, and from results thus obtained, deduced the above factors.

C.—ESTIMATION OF GALACTAN.

Bring 3 to 5 grams of substance free from fatty matters into a beaker of about 5.5 cm diameter and 7 cm deep, and add 60 cc of HNO_3 of 1.15 sp. gr. and evaporate the solution to exactly one-third of its original volume in a water bath at 94° to 96° C. Let the evaporated solution stand twenty-four hours, then add 10 cc of water and let stand twenty-four hours more, thus allowing the mucic acid to crystallize out. In case of most organic substances the mucic acid will be mixed with more or less impurities, and it is therefore necessary to filter, to wash with not over 20 cc of water, and bring the substance and filter back into the beaker. Add 25 to 30 cc of ammonium carbonate,² and bring the solution near to boiling. The $(\text{NH}_4)_2\text{CO}_3$ solution containing the mucic acid is filtered preferably into a platinum dish, the residue well washed, and the entire solution evaporated to dryness in a water bath, and then acidified with dilute HNO_3 , and stirred with a glass rod. The mucic acid separates out, and after standing one-half hour the material is filtered either onto a tarred filter or into a weighed Gooch crucible, and washed with very little water (10 to 15 cc at most) with 60 cc alcohol and with ether, dried at 100° C. and weighed. After reducing to basis of 1 gram multiply by 1.333, which will reduce mucic acid to galactose. Galactose multiplied by 0.9 will give galactan. In case of sample 3 it will not be necessary to extract with ether or $(\text{NH}_4)_2\text{CO}_3$.

RESULTS OF GALACTAN DETERMINATIONS.

One chemist reports that he could do nothing with the method, and two did not try it. The following are the only results received:

Analysts.	Pure milk sugar.		Blue lupine.	
	Galactan.	Galactose, <i>a</i>	Galactan.	Galactose.
United States Department of Agriculture, W. H. Krug	<i>Per cent.</i> 48.75	<i>Per cent.</i> 54.17	<i>Per cent.</i>	<i>Per cent.</i>
Columbia University, H. C. Sherman	46.95	52.17	13.11	14.57
Massachusetts Station:				
J. B. Lindsey	44.04	48.93	14.85	16.50
E. B. Holland	14.66	16.29

a Fifty is theoretical percentage.

¹ Leitschrift für Angw. Chem. 1896, Hefts 2 and 7, Journal für Landw. 1896.

² Ammonium carbonate. Mix 1 part dry $(\text{NH}_4)_2\text{CO}_3$ with 19 parts water and 1 part NH_4OH .

While the results are fairly good, I am convinced that with a little practice still better ones could be obtained. The method is without doubt as accurate as that for estimating pentosans, and gives us a fairly correct idea of the amount of galactan present.

Recent investigations have shown that the hemi-cellulose galactans, while not so generally distributed as the pentosans, exist in considerable quantities, especially in leguminous plants and seeds. It has seemed wise, therefore, to encourage chemists to become accustomed to the use of the only quantitative method thus far proposed for their estimation.

This method consists in the oxidation of the galactan with dilute nitric acid and the weighing of the resulting mucic acid, $C_4H_4(OH)_4(COOH)_2$. Scheele¹ was the first to recognize that by the oxidation of milk sugar with nitric acid mucic acid resulted. Pasteur found that it was the galactose of the milk sugar that yielded the mucic acid. Kent and Tollens² evaporated 100 grams of milk sugar with 1,200 cc of nitric acid of 1.15 sp. gr. in a water bath to one-third of its volume, and allowed the resulting solution to stand twenty-four hours for the mucic acid to crystallize. The precipitate filtered, dried at 100° C. and weighed was found to yield from 37.5 to 40 per cent of mucic acid. When pure galactose was used a double quantity (74 to 77 per cent) was obtained. Rischbieth Creydt, Hadecke, and Tollens³ further perfected the method and used it for the estimation of galactan in a variety of substances. The method with slight modifications was sent out for trial this year together with samples 3 and 4. The percentage of galactan in each was requested. Sample 3 was pure milk sugar. Sample 4 was finely ground blue lupine seed.

AMENDED AND ADOPTED REPORT OF THE COMMITTEE OF RECOMMENDATIONS IN RELATION TO FOODS AND FEEDING STUFFS.

(1) That the diastase and Maercker methods be adopted as general methods for determination of starch and the Sachsse method for the determination of starch in potatoes and commercial starches.

(2) That the methods on page 62 (7) under (a), (b), and (c), and on page 63 under (e), be made official, and that (d), on page 62, remain a provisional method, and also that for the factors on page 63 the following be substituted:

Weight of hydrazon $\times 0.516 + 0.0104 \div$ by amount of substance taken = furfurol.

Furfurol $\times 1.84$ = pentosans.

Pentoses $\times 0.88$ = pentoses.

Furfurol $\times 1.65$ = xylan.

Furfurol $\times 2.03$ = araban.

H. J. WHEELER,

For Committee of Recommendations of Reporters.

The PRESIDENT. Are there any papers relating to this subject?

Mr. LINDSEY. I have the title of a paper by myself and Mr. Holland on the distribution of galactans in agricultural plants which I would like to offer. The paper will be presented to the meeting of the Agricultural Colleges and Experiment Stations and published in the proceedings of that association.

¹ Opuscula Chemica et physica, Leipsig, 1789, p. 111.

² Ann. Chem., 227, p. 221.

³ Landw. Vers. Sta. 39, p. 401.

THE PHLOROGLUCIN METHOD FOR THE ESTIMATION OF PENTOSANS.

By J. B. LINDSEY and E. B. HOLLAND.

Counciler¹ has suggested that instead of phenylhydrazin, phloroglucin be employed for the precipitation and estimation of furfural obtained by the distillation of various substances with dilute hydrochloric acid. Kruger and Tollens² have further studied and perfected the method, and recommend it as reliable for the estimation of pentosans in various coarse fodders, grains, and vegetables. The phloroglucin, just as the phenylhydrazin method, is based on the fact that the pentosans (araban, xylan, etc.) differ from other carbohydrates in that they yield furfural instead of levulinic acid, upon digestion with moderately dilute hydrochloric or sulphuric acids. The first step necessary in both processes for a quantitative estimation is the conversion of the pentosans into furfural, and its separation from the resulting by-products.

Phloroglucin method described.—Three grams of the material are brought into a 10-ounce flask together with 100 cc of 12 per cent hydrochloric acid (sp. gr. 1.06) and several pieces of recently heated pumice stone. The flask placed upon wire gauze is connected with a Liebig condenser and heat applied, rather gently at first, and so regulated as to distill over 30 cc in ten to fifteen minutes from the time that boiling begins. The 30 cc driven over are replaced by a like quantity of the dilute acid by means of a separatory funnel, and the process so continued as long as the distillate gives a pronounced reaction with anilin acetate on filter paper (a few drops of anilin in a little 50 per cent acetic acid). To the completed distillate is gradually added a quantity of phloroglucin³ dissolved in 12 per cent hydrochloric acid, and the resulting mixture thoroughly stirred. The solution first turns yellow, then green, and very soon an amorphous greenish precipitate appears, which grows rapidly darker, till it finally becomes almost black. The solution is made up to 500 cc with 12 per cent hydrochloric acid, and allowed to stand over night. In case there is but very little furfural in the substance tested, and the resulting distillate consequently small, it is best to add sufficient 12 per cent hydrochloric acid to the distillate before adding the phloroglucin solution, so that upon the addition of the latter solution the resulting mixture will contain approximately 500 cc. The amorphous black precipitate is filtered into a tared Gooch crucible through an asbestos felt, washed with 100 cc of water, dried to constant weight by heating three to four hours at 100° C., cooled, and weighed, the increase in weight being reckoned as phloroglucid. To calculate the furfural from the phloroglucid,⁴ use the following table:

Total weight of phloroglucid obtained:	Total weight of phloroglucid obtained:
0.2 divided by 1.82 = furfural.	0.34 divided by 1.911 = furfural.
.22 divided by 1.839 = furfural.	.36 divided by 1.916 = furfural.
.24 divided by 1.856 = furfural.	.38 divided by 1.919 = furfural.
.26 divided by 1.871 = furfural.	.40 divided by 1.920 = furfural.
.28 divided by 1.884 = furfural.	.45 divided by 1.927 = furfural.
.30 divided by 1.895 = furfural.	.50 ÷ divided by 1.930 = furfural.
.32 divided by 1.904 = furfural.	

Furfural divided by grams substance taken $\times 1.84$ = pentosans.

Furfural divided by grams substance taken $\times 1.65$ = xylan.

Furfural divided by grams substance taken $\times 2.03$ = araban.

¹Chemikerztg, 1894, No. 51.

²Zeitsch, für Ang. Chem., 1896, Heft II.

³Dissolve twice as much dry phloroglucin as furfural expected in about 50 cc of 12 per cent hydrochloric acid. Bring the hydrochloric acid into a water bath, and stir thoroughly till the phloroglucin goes into solution.

⁴The phloroglucin is a complex substance of uncertain formula. It contains 63 to 64 per cent of carbon, and from 3.6 to 4.2 per cent of hydrogen. The factors for calculating the amount of furfural from the phloroglucid were obtained after experimenting with known amounts of pure furfural and phloroglucin.

The amount of pentosans was estimated by both the phenylhydrazin and the phloroglucin methods in the following substances:

Name of substance.	By phenyl- hydrazin.	By phloro- glucin.
	<i>Per cent.</i>	<i>Per cent.</i>
English hay	21.28	22.50
High-grown salt hay.....	25.64	25.74
Branch grass	24.65	26.43
Low-meadow fox grass.....	27.98	27.91
Buffalo gluten feed	16.45	16.00
Lupine seeds.....	9.42	9.64

With two exceptions the two methods show very closely agreeing results. We propose to still further compare the methods in the near future. The phloroglucin method, on account of its greater simplicity, is much to be preferred.

The PRESIDENT. Are there any other papers on this subject? If not, the discussion of the report is in order. Has anyone any remarks to offer on the report just presented?

Mr. HUSTON. I do not think that report, which contains the result of a good deal of hard work, ought to pass without an expression of appreciation. I think there is no one here who knows anything about it or is capable of discussing it, and that is the reason it is not discussed. We appreciate how very hard the work is. It seems to me it is deserving of the very heartiest encouragement on the part of this association.

Mr. WILEY. I would like to say, Mr. President, that in our laboratory we have compared the method described by the reporter of collecting, filtering, and weighing the precipitate of furfurollhydrazon. We found it difficult to separate this precipitate easily from the beakers in which it is formed. It is very sticky, and requires a great deal of care to detach it from the sides of the beaker. This led Mr. Krug to introduce a modification of the process, which consists in dissolving the residue of the precipitate in alcohol instead of trying to collect it altogether on a filter. I would like Mr. Krug to explain this method.

Mr. KRUG. In our laboratory we make no attempt to collect the precipitate which sticks to the beaker. It is washed with the prescribed amount of water, which is in turn passed through the filter tube. After drying, the precipitate is dissolved in warm alcohol and the solution collected in a weighed dish, the alcohol recovered, the residue dried at 60° and weighed.

Mr. WILEY. It seems to me that the members of this association do not appreciate the importance of these investigations. We have seen in the last few years a great deal of work done in this country on digestion experiments. In my own opinion the value of digestion experiments depends first of all, upon our knowledge of the material digested. Why should we be so careful to do all this work on digestion experiments without informing ourselves first of the nature of the materials which

are used in the experiments? For this reason I heartily indorse what the reporter has said about the apathy existing with regard to this class of investigations. The remark the reporter made at the beginning of his address had much truth in it. He said he could read his paper now as well as at any time, for there was no one here who wanted to hear it. I hope what he said may induce the members of this association to take a greater interest in this class of investigations, for I believe it is along these lines we are to make our great progress. This is a line of work that demands our attention, and which ought to have it, and I hope future reporters may not have an opportunity to go upon the stage with a justification of the remark with which our distinguished friend introduced his address.

Mr. KILGORE. I would like to say just a word or two upon this subject. I think it is not exactly true, as Dr. Wiley says, that we do not have an interest in this work, because a great many do have an interest in it, but I think generally in the experiment stations the workers are engaged in a chemical line, and we have a certain amount of fertilizer work to do together with a great deal of miscellaneous work which must be done, and after this is done we have little time or energy to take up anything else; so I do not think it a matter of lack of interest, but rather lack of time and pressure of other work which we are forced to do, and I think this association could do no better than in some way to impress upon the authorities who have control of the time of station workers the importance of this class of work, and of putting more time upon it and less time upon regular routine, fertilizer, and miscellaneous work. I should like very much to see something of this kind done, because the workers with whom I have come in contact would like to do this work, but it requires time.

Mr. WILEY. I think Dr. Kilgore is quite right in that respect, because I know that in a great many of the stations the time of the chemists is taken up almost entirely, but I think there are others where there might be some little time devoted to these investigations. We have some things in our laboratory new to you, and if at any time during the progress of the meeting members could visit it we would be glad to have them do so.

The PRESIDENT. The association certainly will be glad to avail itself of Dr. Wiley's kind invitation to visit his laboratory and inspect points of interest.

We were considering the potash report. We have not discussed this report owing to the absence of the reporter at the time it was called upon. Has anyone any remarks to offer? Are there any papers on the subject of potash?

I would like to state for the benefit of those not here after the noon recess that it was moved by Dr. Wiley and the motion adopted that Messrs. Winton and Wheeler, who had been connected with the potash work for the past year or two, be requested to prepare a reply

to some of the recent criticisms that have been made on the Lindo-Gladding method, and particularly, I believe, with regard to the article which Mr. Winton spoke upon this morning.

Mr. WINTON. I neglected to call attention this morning to some samples prepared by the German Association of Experiment Stations and distributed around at the different stations for work.

Mr. KILGORE. The work two years ago on phosphoric acid, when we had the cooperation of some three or four German chemists, we had the criticism made from the director of the chemical station at Norway that with so small a quantity of material that we used in the analyses of phosphates it would be impossible for them to get anything like accurate or concordant results on materials they worked upon. The materials they used were so much larger that they could not work upon the small quantity we employed and get any results.

The PRESIDENT. Are there any further remarks on the subject of potash? If not, the next thing in order will be the report of the committee on the recommendations of the reporter on nitrogen.

Mr. WHEELER. The committee is ready to report on nitrogen, but not on potash. It wishes to call attention to the fact that in the case of nitrates the results were not fully satisfactory.

Mr. LINDSEY. What method do you use in the nitrates?

Mr. WHEELER. The present modified Kjeldahl method, and I think that can be used for that purpose, according to our present regulations.

The PRESIDENT. You have heard the report of the committee. Are there any remarks on the subject?

Mr. WILEY. It seems to me that if the investigations of the reporter indicate that this method—the Ulsch-Street—is not applicable for pure nitrates, the reason for adopting it for nitrates mixed with other bodies ought to be plainly stated, so common people could understand them.

Mr. WHEELER. I feel sure that the association should understand all the circumstances connected with this recommendation. It may be that the trouble is owing to some little thing about the method which is not understood, but which can be understood later and which can be obviated. There is a feeling upon the part of some that on account of its rapidity and on account of the small amount of error when the percentage of nitrates is low it might be adopted for mixed goods. There are, I think, very good reasons why we ought to adopt, even for such goods, a method which will give first-class results.

Mr. VAN SLYKE. Another point which was brought up in the committee was this: The position which the committee takes is much more critical than that taken by the association; and although the results presented on the determination of nitrate of soda do not appear to be what we call first class, yet, compared with the results presented a few years ago for the determination of nitrates, they are entirely satisfactory, and there are not as great discrepancies in the form of nitrate of soda as we found when the other methods were adopted. The

committee is more conservative than the association was, as a whole, when these other methods were adopted.

Mr. WILEY. I believe the method is good enough for nitrates in general, and that is the reason I do not want to see the committee recommend any recession in this method. If some of us are not able to get good results as analysts, I do not think we ought to condemn the method. Why ought we make this discrimination? I approve of the method, and therefore it seems to me that unless it can be proved that it is not a true method we ought not to let go the hold we have taken unless the committee could propose a better method for nitric nitrogen. Do not let us throw down until we are ready to build up. If it is good enough for nitric nitrogen and mixed fertilizers it is good enough for nitric nitrogen and unmixed fertilizers. I think the committee is a little bit too conservative right here.

Mr. STREET. I would like to repeat something I said during the meeting of the committee. It seems to me that in a method of this kind the experiences of workers who have been using it for a long time ought to pass for more than those who have tried it for the first time. Any new method will probably give trouble the first time. We have used it continuously for five years. The first year we used it as a check method, and it gave perfect satisfaction. Since then we have used it exclusively. I agree perfectly with Dr. Wiley in urging that we retain the method until we get a better one.

Mr. KILGORE. The method as it stands right now is not an official method. I infer from Dr. Wiley's remarks that he considers it an official method. It is merely a provisional method, as I consider it now. Looking at these results on the pure soil and at those by the Keldow method, the question arises, Which gives the better results? If we look at the results by the Kjeldahl method, they are very good; if we look opposite, and see the man who gets good results with the Keldow method, we will see, also, that he gets good results by the other method. It appears to be rather a matter of the analyst.

Mr. DAVIDSON. I think the method can be used for pure goods if well distilled, as far as you can distill. It may take forty minutes and it may take more than forty minutes. I think that as the method is now, forty minutes ought to be sufficient time, but distill until all has come off.

Mr. STREET. I said "distill from thirty-five to forty minutes—nearly to dryness."

Mr. WILEY. Would it not be well to announce the rule in regard to those who have a right to vote on this? Most of us have no votes in this matter, and there might be some who may misunderstand the matter. I would suggest the president announce those who have a right to vote in this matter.

The PRESIDENT. There shall be one vote for each institution connected with the fertilizer work.

Mr. WHEELER. I would like to say that the committee had one other point to recommend which bears upon this, and that is, that the Ulsch method be tried, and be referred to the reporter for next year.

Mr. LINDSEY. I would certainly object to the recommendation of the committee that the method be adopted for nitrogen nitrates and mixed goods if it can not recommend it for nitrogen and nitrates, that is, nitrate of soda. If it would determine one right it would determine others. My opinion is that the method is safe to recommend for a method of determination of nitrates; but if the committee does not feel it is safe for unmixed goods, then it should not be adopted for either.

Mr. FREAR. I think we shall find from time to time our special fertilizers will increase in numbers. I notice that tendency in our own State. We have an increased tendency toward the use of nitrates, so we may run upon a snag there. I have always felt that we ought not to be guided by the logic of a method, but by what we find to be the effect in practice concerning the ease with which it can be carried out by the young men upon whom we have to place the burden of the work in our laboratories. For that reason I am inclined to be on the side of the committee's recommendation that we do not say the method is a bad one for the determination of nitric nitrogen in pure goods, but we have found such difficulty in the determination of the nitric nitrogen, even by fairly experienced analysts who do not have occasion to use this method every day, that we hesitate to make a general recommendation of the method for the use of all men who have this fertilizer control work to do. There are only a few fertilizers in the United States where the determination of nitric nitrogen can be carried out. In our own State we make a certain number upon each fertilizer. The character of the determinations to be made is fixed by the department of agriculture of the State, which pays for the work, and it is ready to pay for only so many determinations; and, it goes further, it is willing to print only so many, no matter how many we contribute. So we soon reach a limit. I think, therefore, we will find there will be hereafter, as in the past, a great many analysts who would have occasion to use this method only infrequently, and they would have the same difficulty as heretofore, unless we can strike upon some modification.

Mr. BIGELOW. I do not think many of our association methods or many of the methods used in analytical chemistry are of much value in the hands of others than experts, and I do not think a good method should be abandoned because it requires some experience. If there are those who have to make determinations of nitric nitrogen who can not make them by this method on account of having so little work to do in this line, I think it would be well for them to use a method they can, and give those who are expert an opportunity to use a more perfect method.

Mr. FREAR. When I spoke of experts I did not mean an expert in general, but an expert on the subject in general. I do not know where

the trouble is, but they have tried several modifications with no better result. There is no reason why it should not be continued as an optional method for the determination of nitrogen in nitrates. Do I understand it is the intention of the committee to eliminate altogether from the list the methods which may be used, or simply to make it official for mixed goods and let it stand optional for pure nitrates?

Mr. WHEELER. The recommendation was to make it an official method in the case of the determination of nitrates and mixed goods.

Mr. FREAR. And make no mention of its application to other determinations?

Mr. WHEELER. Not at all. I will say that one of my assistants, who is an excellent analyst and has been working in the laboratory for eight years, failed to get good results, and for that reason asked the reporter not to report his results. They were as good as some of the results here, but he was not satisfied.

Mr. STREET. In our own case during the last season we have had to make over 200 determinations of nitrates, and I am sure there are stations which have to make a corresponding number. We can use this method all right, and I can not see why the experience of those who make an occasional determination should rule out our experience where we have made hundreds of them. Why can not we have this method adopted as a regular method? We can use it. Give us the opportunity of using this method and of feeling we are official.

Mr. WILEY. I do not want to make an amendment, but I do not want to put ourselves in a hole. I do not want this society to go before the world recommending a method for the determination of a matter which is very small in quantity because a little difference will not count, while they are afraid to recommend it for a larger amount where the difference would count. That is not right. Let us be fair with the world and ourselves. If the committee think it is not good enough to use, let it say so.

Mr. LINDSEY. To get out of this difficulty, would it not be wise for the committee to reconsider this point before it makes a final report?

Mr. VAN SLYKE. I believe there is no motion before us now, and I will bring the matter into specific form. I move that the Ulsch-Street method stand as it does now and be referred to the reporter on nitrogen for next year to test with special reference to nitrates in pure goods.

Mr. WHEELER. I second that motion.

Motion carried.

Mr. WHEELER. The second recommendation of the committee, that the heretofore so-called Fassbender method be referred to the reporter on nitrogen for next year for further investigation, and that he look into the matter of attaching a correct name to the method.

Recommendation adopted.

The meeting was then adjourned until Saturday morning.

SECOND DAY.

SATURDAY—MORNING SESSION.

The association met at 10 o'clock, and the president called for the report of the committee on recommendations.

Mr. WHEELER. I will read at the present time the report of the committee on recommendations so far as it has agreed on the points which have been referred to it by the reporters and by the association.

The committee's report was read, and after a brief discussion and making a few changes was adopted.

The PRESIDENT. The next thing in order is the consideration of the report on phosphoric acid.

Mr. McDONNELL. I would say that some of the reports did not reach me until two days before the meeting, and I was considerably rushed in tabulating them. I hope the association will bear with me and pardon the number of defects in my report.

REPORT ON PHOSPHORIC ACID.

By H. B. McDONNELL.

Following the usual custom, the reporter on phosphoric acid sent a circular letter early in the year to a number of chemists who were known to be interested in the determination of phosphoric acid in fertilizers, asking for their cooperation in the work.

Chemists representing twenty laboratories signified their willingness to take part in the work.

Samples were accordingly sent, and with them the following letter of instructions:

COLLEGE PARK, MD., *February 20, 1896.*

DEAR SIR: I send you herewith by express three samples for testing methods for determination of phosphoric acid for the Association of Official Agricultural Chemists.

The association last year directed the reporter on phosphoric acid to make a further test of the volumetric method and also to include in the work the magnesia or citrate method.

For this purpose three samples have been selected as follows:

No. 1. Ground South Carolina rock.

No. 2. Dissolved South Carolina rock.

No. 3. Complete fertilizer, made by mixing 150 samples of miscellaneous "complete" fertilizers.

You are requested to determine total P_2O_5 in all, and soluble and insoluble in Nos. 2 and 3 by the following methods:

1. Volumetric method as modified by reporter.

2. Volumetric method (Chem. Bul. No. 46, U. S. Dept. of Agr.).

3. The official method (Chem. Bul. No. 46, U. S. Dept. of Agr.).

4. A magnesia or citrate method.

5. A magnesia or citrate method as carried out by Glaser (American Fertilizer, Sept., 1895).

6. By weighing the ammonium-phosphomolybdate as carried out by Gladding (Jour. Amer. Chem. Soc., Jan., 1896).

1. The reporter finds that the volumetric method as published is capable of being further simplified and recommends the following method:

Dissolve the phosphate as in the official method, using preferably 40 cc strong HNO_3 with a little HCl , make up to definite volume, take an aliquot portion corresponding to 0.4 gram substance; if over 5 per cent P_2O_5 be present half this amount. Add ammonia in slight excess, then HNO_3 in slight excess and warm to about 50°C . and add the regular molybdic solution in excess, using 75 to 80 cc for each 0.1 gram P_2O_5 present, and in no case less than 10 cc; stir well, let stand for about thirty minutes at 40 to 50°C ., stirring several times at intervals; filter through good filter paper about 9 cm in diameter, on ordinary funnel without suction; wash precipitate with water at ordinary temperature, first by decantation and then on the filter; when filtrate measures about 250 cc test filtrate, as it drops through the funnel, with a small strip of litmus paper; wash once more, stirring the precipitate with the water jet, and test again. When two successive tests show no acidity of filtrate the washing is sufficient. Remove filter and contents to beaker; add standard KOH in excess and stir till precipitate is dissolved and titrate with standard HNO_3 , using phenolphthalein as indicator. The standard solutions are made as directed on page 14, Bulletin 46, Chemistry Division, United States Department of Agriculture.

2 and 3. As in Chemical Bulletin No. 46, United States Department of Agriculture.

4. Citrate method:

Make solution of phosphate according to one of the official methods. Take aliquot corresponding to 0.4 gram for ordinary work or twice this amount for ordinary "insolubles;" make alkaline with ammonia and add ordinary ammonium citrate till clear; cool rapidly, if necessary, and add magnesia mixture slowly from a burette, stirring vigorously; use from 10 to 30 cc magnesia mixture. The latter is sufficient for 30 per cent P_2O_5 on the basis of 0.4 gram substance. Add ammonia in sufficient amount to insure the presence of 3 per cent of free NH_3 in the solution; stir well every half hour and filter after two to five hours. The longer time is required when the amount of P_2O_5 is small. Wash and ignite in the ordinary way. If white residues are desired the last washing should be made with a solution containing $2\frac{1}{2}$ per cent NH_3 and about 40 per cent NH_4NO_3 .

Wiley and Runyan (Jour. Amer. Chem. Soc., July, 1895) recommend the addition of a phosphate solution of known strength when determining P_2O_5 in insoluble residues, etc., which contain less than 5 per cent P_2O_5 , so that the total amount present in solution will be equivalent to 10 per cent. This method should be given a trial with the "insolubles" of samples Nos. 2 and 3.

5. Citrate method as executed by Glaser:

(a) 500 grams citric acid dissolved in 500 cc of water.

(b) Magnesia mixture is prepared by using instead of magnesium chlorid an equivalent amount of magnesium sulphate.

Dissolve the phosphate and take aliquot in the ordinary way except for insolubles; filter solution from insoluble residue and use the whole amount; add strong ammonia in excess and citric acid solution from a burette till the solution is just clear; note the amount and add as much more; cool the solution. Be sure same is alkaline and add twice as much magnesia mixture as citric acid. If more than 5 cc of citric acid have been used the magnesia mixture may be run in quickly. Stir vigorously for several minutes and let settle till liquid becomes clear and stir once again. Filter after one to one and one-half hours, and wash with $2\frac{1}{2}$ to 3 per cent ammonia. For the last washing use a few drops of this solution made strong with ammon. nitrate.

6. The method of Gladding is capable of giving excellent results, and it is the opinion of the reporter that this method will prove of more value to the association than any of the citrate methods. It is as follows:

Make solution and take aliquot in ordinary way; add 25 cc ammonia sp. gr. 0.9 and nitric acid sp. gr. 1.42 till acid; place beaker and contents in water-bath at 50°C .; add ordinary molybdate solution from a burette at the rate of three drops per second, with constant stirring; 50 cc may be added in five minutes. After about 10 cc in excess has been added allow beaker to remain in the bath for ten minutes and filter through a weighed filter paper (reporter suggests a Gooch crucible). Test filtrate, without the washings, by adding 5 cc molybdic solution and replacing in bath for ten minutes. Wash precipitate with nitric acid and water 1:100; three generous washings by decantation and three on the filter and a final washing with water are sufficient. Drain paper and contents for a few minutes on filter or blotting paper and dry to constant weight in oven at 105°C . Use double-walled oven containing dilute glycerol sp. gr. 1.16 (or saturated brine solution). Start drying on the upper shelf and end on lower shelf of oven. The drying requires three or four hours. The precipitate contains 3.76 per cent P_2O_5 .

If you do not care to do all the work outlined above and can undertake part it will be appreciated, as will also any further trials of the methods or modifications that you may care to undertake. In reporting results give results from same flask only on the same horizontal line and use letters *a*, *b*, *c*, etc., to indicate same. One flask

of solution will be sufficient to make one test by several of the methods, and in this way much time may be saved.

In reporting results please give approximate date of making the analyses. It is desirable that the work be done within three months after receiving the samples. Please give full notes, etc., of variations from published methods and send report of results at least ten days before the next meeting of the association.

Yours, truly,

H. B. McDONNELL, *Reporter.*

Reports have been received of the work of seventeen chemists, representing eleven laboratories. Most of the chemists reported duplicate or triplicate results; some as many as nine determinations by the same method on one sample. For lack of space and to facilitate reference only the means are given in the tables. The results reported by the various analysts are as follows:

TABLE I.—TOTAL PHOSPHORIC ACID.

Analyst.	Official gravimet- ric.	Volumet- ric.	Modified volumet- ric.	Gladding	Citrate.	
					(a)	(b) Glaser's.
<i>Sample No. 1.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
C. L. Hare, Alabama.....	27.50	27.75	27.80
J. H. Pratt, Florida.....	27.75	27.92	27.94	27.79
L. H. Merrill, Maine.....	⁰ 28.47	28.52	28.69
C. Glaser, Baltimore, Md.....	$\left\{ \begin{array}{l} {}^0 128.76 \\ {}^2 28.41 \end{array} \right\}$	⁰ 28.38	$\left\{ \begin{array}{l} {}^0 3 28.72 \\ 428.06 \\ {}^0 5 27.94 \end{array} \right\}$	28.35
F. B. Bomberger, Maryland.....	27.40	$\left\{ \begin{array}{l} {}^0 6 27.78 \\ {}^0 7 28.14 \\ 8 28.16 \end{array} \right\}$
W. Skinner, Maryland.....	27.85	27.35	27.30	$\left\{ \begin{array}{l} {}^0 6 27.44 \\ 7 27.94 \end{array} \right\}$	$\left\{ \begin{array}{l} {}^0 6 27.72 \\ 7 27.92 \end{array} \right\}$
F. P. Veitch, Maryland.....	27.82	27.43	27.48	27.65	28.00	28.11
W. M. Allen, North Carolina.....	28.00
H. K. Miller, North Carolina.....	27.95	27.84
B. W. Kilgore, North Carolina.....	27.90
C. B. Williams, North Carolina.....	27.95
M. S. McDowell, Pennsylvania.....	⁰ 28.43	27.04	28.08	28.41	28.22	28.47
E. G. Runyan, District of Columbia...	27.55	27.62	27.63	$\left\{ \begin{array}{l} {}^0 26.63 \\ {}^0 26.81 \end{array} \right\}$	27.63	27.59
C. H. Jones, Vermont.....	27.89	$\left\{ \begin{array}{l} {}^0 9 26.74 \\ 27.42 \end{array} \right\}$	27.38	27.38	28.01	28.14
F. W. Woll, Wisconsin.....	28.16	27.59	28.12
J. P. Street, New Jersey.....	27.68	27.54	27.70	27.79	28.04
Number averaged.....	11	12	10	4	8	9
Average.....	27.87	27.66	27.68	27.88	27.91	28.11
<i>Sample No. 2.</i>						
C. L. Hare, Alabama.....	15.50	15.74	15.81
J. H. Pratt, Florida.....	15.54	15.54	15.55	15.52

⁰ Omitted from average.

¹ Before ignition with HNO₃. Omitted from average.

² After ignition with HNO₃. Omitted from average.

³ Two and one-half hours drying. Omitted from average.

⁴ Four hours drying.

⁵ Four hours drying, wash water milky. Omitted from average.

⁶ Stood 2 hours. Omitted from average.

⁷ Stood 21 hours. Omitted from average.

⁸ Stood 24 hours.

⁹ Obtained when solution made as per (a₁) page 12, official method. Omitted from average.

TABLE I.—TOTAL PHOSPHORIC ACID—Continued.

Analyst.	Official gravimet- ric.	Volumet- ric.	Modified volumet- ric.	Gladding.	Citrate.	
					(a)	(b) Glaser's.
<i>Sample No. 2—Continued.</i>						
L. H. Merrill, Maine.....	15.42	15.66	15.76
C. Glaser, Baltimore, Md.....	$\left\{ \begin{array}{l} {}^0 15.68 \\ {}^2 15.65 \end{array} \right\}$	${}^0 3 16.13$	15.28	15.47
F. B. Bomberger, Maryland.....	${}^4 15.25$	$\left\{ \begin{array}{l} {}^0 5 15.28 \\ {}^0 6 15.54 \\ {}^7 15.63 \end{array} \right\}$
W. Skinner, Maryland.....	15.40	15.35	15.42	${}^{10} 15.23$
F. P. Veitch, Maryland.....	15.37	15.38	15.53	15.44	15.44	15.47
W. M. Allen, North Carolina.....	15.63
H. K. Miller, North Carolina.....	15.63	15.54
B. W. Kilgore, North Carolina.....	15.61
C. B. Williams, North Carolina.....	15.58
M. S. McDowell, Pennsylvania.....	${}^0 16.11$	${}^0 15.95$	${}^0 16.73$	15.92	15.67	15.79
E. G. Runyan, District of Columbia...	15.40	15.34	15.42	${}^0 15.05$	15.44	15.43
C. H. Jones, Vermont.....	$\left\{ \begin{array}{l} {}^0 4 15.34 \\ 15.27 \end{array} \right\}$	$\left\{ \begin{array}{l} {}^0 8 14.66 \\ 15.30 \end{array} \right\}$	15.26	15.41	15.42	15.70
F. W. Woll, Wisconsin.....	15.19	15.44	15.66
J. P. Street, New Jersey.....	15.61	15.55	15.77	15.59	15.57
Number averaged.....	12	11	9	3	7	9
Average.....	15.46	15.47	15.48	15.38	15.55	15.58
<i>Sample No. 3.</i>						
C. L. Hare, Alabama.....	11.02	11.00
J. H. Pratt, Florida.....	11.41	11.41	11.42	11.42
L. H. Merrill, Maine.....	11.58	11.52	11.58
C. Glaser, Baltimore, Md.....	11.39	${}^0 12.12$	11.74	11.45
F. B. Bomberger, Maryland.....	11.23	$\left\{ \begin{array}{l} {}^5 11.16 \\ {}^6 11.25 \\ {}^7 11.35 \end{array} \right\}$
W. Skinner, Maryland.....	11.31	11.32	11.35	11.31	11.34
F. P. Veitch, Maryland.....	11.45	11.20	11.25	11.45	11.34	11.34
W. M. Allen, North Carolina.....	11.53
H. K. Miller, North Carolina.....	11.60	11.48
B. W. Kilgore, North Carolina.....	11.49
C. B. Williams, North Carolina.....	11.46
M. S. McDowell, Pennsylvania.....	11.89	${}^0 12.14$	${}^0 12.23$	11.70	11.59	11.60
E. G. Runyan, District of Columbia...	11.44	11.34	11.39	11.07	11.28	11.57
C. H. Jones, Vermont.....	$\left\{ \begin{array}{l} {}^0 8 11.29 \\ 11.31 \end{array} \right\}$	$\left\{ \begin{array}{l} {}^0 8 10.28 \\ 11.34 \end{array} \right\}$	11.43	11.60	11.38	11.48
F. W. Woll, Wisconsin.....	11.40	11.18	11.63
J. P. Street, New Jersey.....	11.57	11.54	11.54	11.47	11.46
Number averaged.....	13	10	9	5	8	9
Average.....	11.45	11.38	11.36	11.51	11.41	11.47

⁰ Omitted from average.¹ Before ignition with HNO₃. Omitted from average.² After ignition with HNO₃. Omitted from average.³ End point not distinct. Omitted from average.⁴ Stood 2 hours at 50°.⁵ Stood 2 hours. Omitted from average.⁶ Stood 21 hours. Omitted from average.⁷ Stood 24 hours.⁸ Obtained when solution made as per (a₄), page 12, official method. Omitted from average.

TABLE II.—SOLUBLE PHOSPHORIC ACID.

Analyst.	Official gravimetric.	Volumetric.	Modified volumetric.	Gladding.	Citrate.	
					(a)	(b) Glaser's.
<i>Sample No. 2.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
C. L. Hare, Alabama	12.49	12.41
J. H. Pratt, Florida	12.50	12.55	12.44
L. H. Merrill, Maine	12.56
C. Glaser, Baltimore, Md	{ ⁰ 12.41 ¹ 12.54}	³ 12.84	12.32
W. Skinner, Maryland	12.37	³ 12.36	12.51	12.48
F. P. Veitch, Maryland	12.58	12.37	{ ⁰ 12.66 12.58}	12.62
M. E. McDonnell, Pennsylvania	12.80	12.93	12.81	12.48	12.73	12.61
M. S. McDowell, Pennsylvania	12.81	12.89	13.00	12.77	12.49	12.60
E. G. Runyan, District of Columbia	12.53	12.41	12.48	⁰ 11.80	12.60	12.44
C. H. Jones, Vermont	⁰ 11.63	⁰ 11.78	⁰ 11.57	⁰ 11.84	⁰ 11.65	⁰ 11.73
Number averaged	9	4	8	3	5	6
Average	12.62	12.65	12.63	12.63	12.59	12.48
<i>Sample No. 3.</i>						
J. H. Pratt, Florida	5.26	5.28	5.23
L. H. Merrill, Maine	5.31
C. Glaser, Baltimore, Md	5.21	5.56	⁴ 5.52	5.20
W. Skinner, Maryland	5.90	5.82	5.86	5.70
F. P. Veitch, Maryland	5.79	5.71	5.74	5.82
M. E. McDonnell, Pennsylvania	5.83	6.05	6.09	5.65	5.65	5.73
M. S. McDowell, Pennsylvania	5.84	5.85	5.90	5.80	5.60	5.66
E. G. Runyan, District of Columbia	5.70	5.76	5.84	5.44	5.46	5.79
C. H. Jones, Vermont	⁰ 4.77	⁰ 5.03	⁰ 5.07	5.41	⁰ 4.64	⁰ 4.98
Number averaged	8	4	7	5	5	6
Average	5.60	5.84	5.76	5.56	5.63	5.55

⁰ Omitted from average.¹ Before igniting with HNO₃.² After igniting with HNO₃. Omitted from average.³ End point not distinct.⁴ Darkening around edge.⁵ Four hours drying, wash water milky. Omitted from average.

TABLE III.—INSOLUBLE PHOSPHORIC ACID.

Analyst.	Official gravimetric.	Volumetric.	Modified volumetric.	Gladding.	Citrate.		
					(a)	(b) Glaser.	Runyan and Wiley.
<i>Sample No. 2.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
C. L. Hare, Alabama	0.55	0.47
J. H. Pratt, Florida5454	0.53
L. H. Merrill, Maine33
C. Glaser, Baltimore, Md	{ ⁰ .55 .66}5149
W. Skinner, Maryland Agricultural College73	0.69	.7252
F. P. Veitch, Maryland Agricultural College75	.76	1.76	0.64	² 0.63	³ .82

⁰ Omitted from average.¹ Stood $\frac{1}{2}$ hour at 40 to 50° before filtering.² Stood 20 hours before filtering.³ Stood 8 hours before filtering.

TABLE III.—INSOLUBLE PHOSPHORIC ACID—Continued.

Analyst.	Official gravi- metric.	Volu- metric.	Modified volu- metric.	Gladding.	Citrate.		
					(a)	(b) Glaser.	Runyan and Wiley.
<i>Sample No. 2—Continued.</i>							
M. E. McDonnell, Pennsyl- vania.....	<i>Per cent.</i> .64	<i>Per cent.</i> .63	<i>Per cent.</i> .63	<i>Per cent.</i> .61	<i>Per cent.</i> .52	<i>Per cent.</i>	<i>Per cent.</i>
M. S. McDowell, Pennsyl- vania.....	.67	.70	.69	.74	.56	.68
E. G. Runyan, United States Department of Agriculture.	.54	.53	.54	0.48
C. H. Jones, Vermont.....	.41	.47	.43	.33	.51	.42
Number of analysts....	10	6	9	4	4	6	1
Average58	.63	.59	.58	.56	.58	.48
<i>Sample No. 3.</i>							
C. L. Hare, Alabama.....	2.28	2.31
J. H. Pratt, Florida.....	2.45	2.49	2.43
L. H. Merrill, Maine.....	2.39
C. Glaser, Baltimore, Md.....	2.22	2.40	2.24
W. Skinner, Maryland Agri- cultural College.....	2.39	2.27	2.29	2.33
F. P. Veitch, Maryland Agri- cultural College.....	2.26	2.25	⁰ 2.25 2.21	2.16	12.10	⁰ 2.07
M. E. McDonnell, Pennsyl- vania.....	2.62	2.66	2.63	2.48	2.63	2.67
M. S. McDowell, Pennsyl- vania.....	2.63	2.59	2.55	2.37	2.47	2.42	2.38
E. G. Runyan, United States Department of Agriculture.	2.26	2.24	2.24	2.26	2.47
C. H. Jones, Vermont.....	2.14	2.37	2.30	2.22	2.27	2.41
Number of analysts....	10	6	9	5	4	7	2
Average	2.36	2.38	2.38	2.30	2.37	2.37	2.43

⁰ Omitted from average. ¹ Stood 20 hours before filtering. ² Stood 8 hours before filtering.

These results are about as good as could reasonably be expected with the volumetric and citrate methods, with which some of the analysts were probably not very familiar.

The results by the official gravimetric method, however, are not as concordant as they should be. In the case of the total phosphoric acid the difference between the highest and lowest results are 1.29, 0.92, and 0.87 per cent on samples Nos. 1, 2, and 3, respectively, being, approximately, 4.50, 6, and 7.50 per cent of the total present.

The results show that the volumetric method is reliable, and that the modifications suggested by the reporter will give results equally as good as the method as published, and is considerably simplified by doing away with the potassium nitrate and nitric acid washes and using only water.

My experiments show that the acid substance which is precipitated more or less from molybdate mixture and which is generally called molybdic acid, but which is probably a hydrated acid ammonium salt of molybdenum, is quite soluble in water—nearly as much so as it is in dilute nitric acid, as the following experiments indicate:

1. Precipitated "molybdic acid" from 100 cc molybdate solution by adding ammonia till nearly neutral and warming; threw the precipitate on a filter and washed with water. After the first washing the precipitate rapidly dissolved, and was

thrown down again as the solution mixed with the first washings. When filtrate measured 450 cc all of the precipitate on the filter had dissolved.

2. Precipitated "molybdic acid" as above; filtered and washed precipitate several times with water; then collected 25 cc of the filtrate and passed it over the precipitate twice; evaporated the 25 cc to dryness and heated in oven to drive off nitric acid, and titrated with the standard alkali (as used in determination of phosphoric acid) and found that 13.2 cc were required to neutralize, equivalent to 52.8 cc for 100 cc of water. This result is evidently too low, because ammonium nitrate was still present and retarded the solution.

3. Precipitated "molybdic acid" as above and brought an equal amount of the precipitate on each of four 10 cm filters till they were nearly full of the precipitate; washed two with water and two with nitric acid wash solution. Four washings (total, 110 cc) of the latter, and five of the former (total, 135 cc) were required to dissolve the contents of each filter.

When it is remembered that in washing with nitric acid we introduce much more acid than is removed in the operation, and which must in turn be washed out with other washes, the saving of time is apparent.

The citrate methods give fair results and, no doubt, would be useful to some. The method as carried out by Glaser shows no advantage over the simpler method.

Citrate methods have a tendency to give low results when small amounts of phosphoric acid are present. The method proposed by Runyan and Wiley for overcoming this difficulty by adding a known amount of phosphoric acid, in the form of a soluble phosphate, to each determination and subtracting it from the final result has been tried by but two analysts. These results do not indicate the necessity for this precaution.

The results on insoluble phosphoric acid are not very satisfactory on sample No. 2. The difference between the highest and the lowest results is 0.42 per cent, which is 73 per cent of the average. On sample No. 3 the results are better. Difference between highest and lowest is 0.39 per cent, which is 16 per cent of the average.

NOTES OF ANALYSTS.

C. L. Hare, Alabama.—The volumetric method, as modified by the reporter, gives satisfaction with all who have tried it here. The only objection is the end reaction with phenolphthalein. It is not sharp enough. This, however, may be overcome in a degree by using small equivalents. The error occasioned by this use of smaller amounts is counterbalanced by the smaller error in reading the end reaction.

The chances for error in the method are much less than in the gravimetric.

I find the ordinary funnel and filter paper holds better than platinum cones with asbestos, or Gooch crucibles with disk of filter paper.

L. H. Merrill, Maine.—I have been unable to get a sharp end reaction with the volumetric method, the color disappearing so gradually that it was impossible to read within a cubic centimeter.

F. P. Veitch, Maryland.—

Official method: Precipitates made white by using a solution of $2\frac{1}{2}$ per cent ammonia, containing 20 per cent ammonium nitrate before ignition.

Volumetric method: Used Gooch crucible with two disks of filter paper for filtering; 225 cc wash water in all cases freed precipitate of acid. Would suggest that for percentages above 18 and below 2 a little longer time in the bath would be advisable; say, eight to ten minutes. Used standard sulphuric acid for titrating.

Modified volumetric method: Filtered same as in method above; 250 cc wash water sufficient in all cases. Results on reporter's samples confirm quite a lot of work on other samples by this modification, which I prefer to the regular volumetric.

Citrate method: Used about twice as much magnesia mixture as required to precipitate the phosphoric acid present. The use of the blast in the final ignition in this method seems to be necessary, otherwise results are too high. Results on insolubles, even after long standing, are a little low.

Citrate method, Glaser's modification: No advantage over other citrate method except in making up citric acid solution. Precipitates came down slowly. Did not stir constantly for half an hour, but for three to five minutes five or six times in three hours.

Gladding method: There is danger of loss in neutralizing the great amount of strong ammonia added. Gooch crucible and two disks of filter paper used for the filtration. Washed more than recommended, which does not seem to be sufficient. Washed about 150 cc, or till neutral to litmus.

B. W. Kilgore.—The volumetric method has proved to be entirely satisfactory in the hands of all the workers in this laboratory.

E. G. Runyan.—Results by weighing ammonium-phospho-molybdate were very unsatisfactory. On filtering through paper and washing as directed, the filter was partially oxydized during the process of drying. When a Gooch was used and the precipitate washed with dilute acid the results appeared to be too high, while, if the final washing was made with water, some of the precipitate always ran through the filter.

J. P. Street.—I had little difficulty with any of the determinations, except with those on sample 1, where I had considerable trouble in getting satisfactory results, especially with the official method.

During the past season I have used a method for phosphoric acid which has given very satisfactory results. It is the Kilgore method with a few modifications. The following is the mode of procedure:

Treat 2 grams of the sample with 5 cc of magnesium nitrate, evaporate, ignite, and dissolve in hydrochloric acid. Take an aliquot portion of the solution (equal to 0.25 gram), 5 cc of concentrated nitric acid, then neutralize with ammonia and clear with a few drops of nitric acid. Heat on the water bath to the temperature of 60 to 65° C., add 50 cc of freshly filtered molybdic solution for each decigram of P_2O_5 present, and digest at this temperature for twenty minutes. Proceed as indicated under (2) (*b*) in Bulletin 46, United States Department of Agriculture, page 14.

I found that solution in nitric and hydrochloric acids would not always answer, so I used the magnesium nitrate uniformly on all samples. Six minutes' heating in many cases will not precipitate the ammonium-phospho-molybdate; the time I indicate—twenty minutes—sufficed in every case. For some reason or other, by this method less water is needed to thoroughly free the precipitate from all traces of acidity; 100 cc were ample in every case. The tabulated comparative results which I inclose were all obtained by this modification.

Comparative results with 326 samples are given, as follows:

Total phosphoric acid, 276 samples—	Per cent.
Average by official gravimetric method.....	10.70
Average by volumetric method.....	10.72
Insoluble phosphoric acid, 50 samples—	
Average by official method.....	2.61
Average by volumetric method.....	2.55

RECOMMENDATIONS.

I would recommend the adoption of the volumetric method, as modified, as one of the official methods. Also that the use of sulphuric or hydrochloric acids be allowed for the standard acid as well as nitric acid.

Amended and adopted report of the committee on recommendations in relation to phosphoric acid.

It is recommended—

(1) That the volumetric method be referred to the reporter for 1897 with the request that it be further investigated with a variety of phosphatic materials.

(2) That on page 14, section 2, under (*b*), the time of digestion be made ten to fifteen minutes instead of six minutes as at present; also that (*b*₁), paragraph 2, in line 6, in the direction for washing, the following be inserted: "Wash five to six times with water, using in all 150 to 250 cc."

(3) That the methods for iron and aluminum in phosphates be referred to the reporter for 1897.

(4) That the methods of determining available phosphoric acid in phosphatic slags be investigated by the reporter for next year.

H. J. WHEELER,

For the Committee on Recommendations of Reporters.

The PRESIDENT. The next thing in order is the presentation of papers. Are there any papers to be presented?

Mr. Huston read a paper on basic slag.

ACTION OF AMMONIUM CITRATE AND CITRIC ACID ON BASIC SLAG.

By H. A. HUSTON and W. J. JONES, Jr.

In view of the various methods proposed for determining the available phosphoric acid in basic slag, it may be of interest to examine the action of the substances used for the purpose, when they are applied under different conditions of time, temperature, quantity, acidity, and concentration.

In the work here reported all digestions were made in closed flasks placed in the digesting apparatus described in Bulletin 54, Indiana Agricultural Experiment Station, and Wiley's Agricultural Analysis, page 142.

The basic slag used was made at Pottstown, Pa., and contained 18.24 per cent total phosphoric acid. It was ground to pass 100-mesh sieve.

Unless otherwise stated, 100 cc of the neutral solvent, 110 cc of solution corrected for basicity, and 2 grams of slag were used. To save space only averages are given. Duplicate determinations were always made, and sometimes the number was increased to four.

As some of the proposed methods attempt to correct the basicity of the slag by the addition of a definite amount of acid before the addition of the citrate solution, two series were made. In one series the slag was treated with neutral ammonium citrate, while in the second series enough citric acid was added to neutralize the basicity of the slag. This amount of citric acid—0.52 gram of citric acid per gram of slag—was determined according to Wagner's method, shaking for one-half hour 5 grams of slag with 500 cc of 1 per cent solution of citric acid. Further investigation of this step might be useful, for we found on adding the above amount of citric acid—0.52 gram per gram of slag—to a fresh portion of slag and shaking one-half hour, that the mixture was not neutral, but contained free acid—only 0.362 gram of acid per gram of slag having been neutralized. When 0.362 gram of citric acid per gram of slag was shaken for one-half hour with a fresh portion of slag, only 0.275 gram of citric acid was neutralized.

Wishing our work to be in some degree comparable with that of Wagner, we used the amount of citric acid indicated by the first titration—0.52 gram of citric acid per gram of slag. But this does not make our solution the same as Wagner's solution, which contains free acid before adding the amount required by the results of the neutralization test.

I.—INFLUENCE OF TIME OF DIGESTION.

[Temperature, 65° C.]

Per cent phosphoric acid dissolved.

Time of digestion.	Series I. Neutral citrate.	Series II. Basicity corrected.
$\frac{1}{2}$ hour	6.90	9.61
1 hour	7.78	10.66
2 hours	8.52	11.18
$3\frac{1}{2}$ hours	9.00	11.60
5 hours	9.15	11.86
$7\frac{1}{2}$ hours	9.02	11.86
10 hours	9.56	11.82

The form of the curve is very similar to the form of the time curve for phosphate of lime in bone products.

In all our work on phosphates we make the time curve first. So far as possible we wish to deal with completed reactions. In this case the solution was practically

complete in five hours. This length of time was therefore adopted as the standard time for the remainder of the work.

Even when the basicity is corrected by the addition of acid, the reaction is far from complete in one-half hour even at a temperature of 65°.

II.—INFLUENCE OF TEMPERATURE.

[Time, five hours.]

Per cent phosphoric acid dissolved.

Temperature of digestion.	Series I. Neutral cit- rate.	Series II. Basicity corrected.
40° C.....	8.10	11.36
50° C.....	9.27	11.46
65° C.....	8.90	11.81
75° C.....	9.12	11.91
85° C.....	8.88	11.81

In seventy-two hours, at room temperature, about 25° C., with neutral solution 7.54 per cent were dissolved.

It will be seen that when the time of digestion is extended to five hours the temperature exerts very little influence. This is not equivalent to saying that the temperature would not exert any influence in the cases where one-half-hour digestions were used.

In its temperature relations slag differs radically from the phosphates of alumina and iron, and also bone, and South Carolina, Florida, or Tennessee rock. It somewhat resembles precipitated phosphate of lime obtained in the acid process of glue making when the latter material is subjected to one-half-hour digestion at various temperatures.

III.—INFLUENCE OF ACID AND ALKALI.

[Time, five hours. Temperature, 65° C. Volume, Series I, 110 cc; Series II, 120 cc.]

Per cent phosphoric acid dissolved.

Amount of acid or alkali added.	Series I. Neutral cit- rate and acid or al- kali added.	Series II. Basicity corrected and acid or alkali added.
2 grams citric acid.....	11.98	12.41
1 gram citric acid.....	11.58	12.18
0.5 gram citric acid.....	10.27	12.07
0.2 gram citric acid.....	9.69	12.04
Nothing.....	8.86	11.85
Ammonia equivalent to 0.2 gram citric acid.....	8.49	11.56
Ammonia equivalent to 0.5 gram citric acid.....	8.05	10.92
Ammonia equivalent to 1 gram citric acid.....	7.25	9.66
Ammonia equivalent to 2 grams citric acid.....	6.20	8.02

Twenty grams of citric acid in 110 cc water dissolve 16.36 per cent (Series I). Twenty-one and four one-hundredths grams of citric acid dissolved in 120 cc water dissolve 16.84 per cent (Series II).

One hundred and ten cubic centimeters ammonia water that would neutralize 20 grams of citric acid dissolve 0.3 per cent (Series I). One hundred and twenty cubic

centimeters of ammonia water that would neutralize 20 grams of citric acid and containing 1.04 grams citric acid dissolve 0.96 per cent.

The form of this curve, on the acid side especially, resembles that of precipitated calcium phosphate. The wide divergence of the two lines near the neutral point is rather remarkable. Points of the same actual acidity in the two lines are not in the same horizontal position, but the upper line is always too high. Whether this be due to the larger quantity of citrate present or to the fact that there is 9 per cent more water present in the case of the high results is a question that is now under investigation.

IV.—INFLUENCE OF THE QUANTITY OF SLAG USED.

[Time, five hours. Temperature, 65°. Volume, Series I, 100 cc; Series II, 110 cc.]

Per cent phosphoric acid dissolved.

Quantity of slag used.	Series I. Neutral cit- rate.	Series II. Basicity corrected.
0.5 gram	12.36	12.41
1 gram	12.37	12.17
2 grams	8.90	11.81
4 grams	3.26	7.37

The curve resembles that for precipitated calcium phosphate, and differs radically from the curves for all other materials that we have examined.

V.—ACTION OF CITRIC ACID ON BASIC SLAG.

Owing to the peculiar action of citric acid on the slag, which has been mentioned above, it was decided to study the action more in detail. The factors investigated were the effect of increased time and temperature on the amount of citric acid neutralized and on the amount of phosphoric acid dissolved. The results of the work appear in the following table:

[Volume, 100 cc of 2 per cent citric acid. Slag used, 1 gram.]

Time of digestion.	Temper- ature.	Neutral- ized citric acid.	Phos- phoric acid dis- solved.
	°C.	Grams.	Per cent.
$\frac{1}{2}$ hour	25	0.666	13.28
1 hour	25	.698	14.25
2 hours	25	.734	14.55
$3\frac{1}{2}$ hours	25	.736	12.50
5 hours	25	.766	13.20
$\frac{1}{2}$ hour	65	.730	12.36
1 hour	65	.780	12.01
2 hours	65	.794	11.04
$3\frac{1}{2}$ hours	65	.824	11.28
5 hours	65	.824	7.64
$\frac{1}{2}$ hour	(¹)	1.124	7.55

¹ Boiling.

The curves show that the amount of citric acid neutralized varies directly with the temperature and the time of digestion. The amount of phosphoric acid remaining in solution at 65° decreases as the time of digestion increases, while at 25° the amount of phosphoric acid remaining in solution increases for a time and then decreases. The direction of the lines seems to indicate a maximum solubility at

this temperature of about 15 per cent at the end of one and one-half hours. At 65° the decrease in the amount of phosphoric acid is very marked, and may be due to a solution of the tetrabasic phosphate and its subsequent conversion into tribasic calcium phosphate, which is alleged to be insoluble in dilute citric acid, or it may be due to a conversion into phosphate of iron and alumina. All acid solutions of basic slag give off sulphureted hydrogen, which makes the reactions even more complex. The amount of citric acid neutralized increases more rapidly than is essential to furnish the acid to convert the tetrabasic phosphate into the tribasic form. Thus, in four and one-half hours, between the end of one-half-hour digestion and the end of five-hour digestions, 0.0472 gram of phosphoric acid becomes insoluble. This is equivalent to 0.128 gram of tetrabasic phosphate of lime. To convert 1 molecule of calcium oxid in this amount of tetrabasic phosphate, or 0.0196 gram calcium oxid, into calcium citrate would require 0.048 gram citric acid. The actual amount of citric acid neutralized in the time given was 0.094 gram.

VI.—COMPARISON OF WAGNER'S METHOD WITH THE OFFICIAL METHOD.

This work was done with sample received from Mr. Voigt. The sample was used in the same state of division. It contained some metallic iron. Total $P_2O_5 = 16.81$ Wagner's reagents; 16.80 official reagents.

Series.	Time	Tem- per- ature.	Wag- ner's re- agents dis- solved.	Offi- cial re- agents dis- solved.	Re- verted by dif- ference, Wag- ner's re- agents.	Re- verted by dif- ference, official re- agents.	Insol- uble by dif- ference, Wag- ner's re- agents.	Insol- uble by dif- ference, official re- agents.	Insol- uble, Wag- ner's re- agents.	Insol- uble, official re- agents.
	Min.	° C.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
No. 1. Wagner's method, acid citrate (5 grams, 500 cc).	30	17½	11.69	11.78	5.12	5.02
			11.90	12.13	4.91	4.67
			11.55	11.80	5.26	5.00
Average			11.71	11.90	5.10	4.90
No. 2. Official citrate, neutral, sp. gr. 1.090 (5 grams, 500 cc).	30	17½	5.86	5.96	10.95	10.84
			5.69	6.18	11.12	10.62
			6.00	6.18	10.81	10.62
Average			5.85	6.11	10.96	10.69
No. 3. Official method, neutral citrate, sp. gr. 1.090 (2 grams, 100 cc).	30	65	5.22	5.06	11.59	11.74
			5.45	5.41	11.36	11.39
			5.26	5.11	11.55	11.69
Average	5.31	5.19	11.50	11.61
No. 3 (b). Official citrate, neu- tral, sp. gr. 1.090 (2 grams, 100 cc).	30	65	5.26	5.00	11.55	11.80
			5.30	5.07	11.51	11.73
			5.23	5.18	11.58	11.64
Average			5.26	5.08	11.54	11.72

The material is not ground finely enough to permit close agreements of duplicates. There seems to be no advantage in Wagner's molybdate or magnesia solution. The filtration and washing of the phosphomolybdate precipitate was very tedious when directly precipitated in the citrate solution. A comparison of set 2 with set 3 shows that a change of volume or quantity of the solvent is of more importance than a change of temperature. If the sample is a fair one a much larger working error than usual must be allowed if the material is to be analyzed in the condition in which it is received.

The PRESIDENT. Are there any remarks?

Mr. GOSS. I would like to say that I consider this work of Professor Huston on the different phosphates as extremely important, not only in the fertilizer work, but also in many other kinds of work—soil work for example—and I for one would be very much pleased to have him publish it. I am interested particularly in this iron and phosphate business.

Mr. WHEELER. I am interested particularly in this iron and aluminum phosphate business. What are we as an association going to do? If we find in goods phosphates which contain as high as 38 per cent, in their pure condition, of reverted acid, which is of no use to plants, what is the use of our present method of determining reverted acid? I feel that we shall have to throw the whole thing overboard. There has got to be an entire revolution. I would like to hear from anybody in this association who has had any experience. I understand that in France they claim good results, but it is surprising how inefficient are these phosphates.

Mr. HUSTON. I have had some experience in the field. The fact is, we must be a little careful about our conclusions in regard to phosphates and aluminum and iron in the field, and you must watch the crop a little. I have made some experiments on corn which show phosphate of aluminum to be the most valuable phosphate yet put on. We have done some greenhouse work on potatoes which shows up very well. We have done some other work on roses on which we have good results. In the first place, the question of reverted phosphoric acid in the natural phosphate of aluminum is nonsense. There is no reverted acid there. There is not any of the material once soluble and no longer so. The fact is, that you can dissolve all of that material in ammonium citrate excepting small traces. It is the same old thing; the same old quantity. You will either use not enough volume or fluid or perhaps not enough spirit of ammonia. That appears to be a very important thing so far as the commercial relations of the thing are concerned. It is between the man who sells and the man who buys it. When that material goes in as a phosphate in the ordinary mixed goods containing not a total of 43 to 48 per cent, but a total of only 13 per cent, then you have not a soluble determination in the mixed goods. You are not working on two drams. You are working upon about one-half a dram, from which you would dissolve in the neighborhood of 42 per cent out of 48 of the total. That is, you come near making a 44 determination of what is left. The trouble is not in the mixed goods. I do not think the question is in the form of the agricultural value. The question is the trouble between the man who sells and the man who buys.

Mr. WHEELER. I would like to ask the gentleman, in consideration of all his results, if he thinks it is desirable for this association, in the light of what knowledge we now have, to continue the use of ammonium citrate in conducting the determinations as we now do for determining the real worth of commercial fertilizers, particularly where a valuation is placed upon the goods?

Mr. HUSTON. Yes; I think it is desirable to continue it just as we do, until we have a good reason for changing it. I believe we had better keep what we have. It is the best we know. We will try to get something better if we can.

The PRESIDENT. Have you had any experience with Wagner's method?

Mr. HUSTON. No; I have not used that at all.

Mr. WHEELER. Every member of this association feels this is one of the most valuable pieces of work undertaken by this association in some time, and I hope Professor Huston will publish his results. I feel that we must have something better just so soon as possible, or take some step to eliminate the errors and difficulties which come in. I hope that his work will be published as soon as may be, so we may have that as a basis upon which to start.

Mr. HUSTON. I referred to the matter of publication last year and stated one reason I did not publish it was because I had no complete analyses of raw phosphates. I asked whether anyone had these, and have not heard from them. I would like a complete analysis of these materials before I publish it. The work is ready to publish as soon as we can get a complete analysis.

The PRESIDENT. The association would be much pleased if this work of Professor Huston's were published in a form that would be accessible to us all.

Mr. McDONNELL. I have a paper by Mr. Veitch which I would like to present.

THE AMOUNT OF WASHING REQUIRED TO REMOVE THE PHOSPHORIC ACID RENDERED SOLUBLE BY DIGESTION WITH AMMONIUM CITRATE AT 65° C.

By F. P. VEITCH.

The amount of washing to be given the insoluble residue from the treatment with ammonium citrate has never been considered by this association, so far as I know, although the subject has been brought to its notice at least twice (Bull. 31, p. 113, and Bull. 35, p. 37, Div. Chem., U. S. Dept. Agr.).

The determination of insoluble phosphoric acid is the determination upon which there is the greatest difference in results among chemists engaged in the fertilizer control. Being desirous of knowing how much this difference may be due to differences in the amounts of wash water used to remove the phosphoric acid rendered soluble by the treatment with ammonium citrate, fifteen samples of fertilizers, representing the commonly occurring goods on our market, were treated as usual until the ammonium citrate was filtered from the residues. The residues were then washed with 100 cc of water at 65° C. These washings were thrown away, the residues further washed until four portions of 100 cc each had passed through the filters; the phosphoric acid was then determined in these four sets of washings, with the following results.

TABLE I.—Per cent of P_2O_5 removed by washing.

No.	Fertilizer.	Removed by each 100 cc of wash water.					Insoluble found, washed to 250 cc.
		Second.	Third.	Fourth.	Fifth.	Total.	
1	Dissolved South Carolina rock.....	0.030	0.025	0.020	0.013	0.088	1.05
2do.....	.033	.013	.043	.020	.109	.80
3	Dissolved South Carolina rock and potash ..	.085	.023	.013	.010	.131	.94
4do.....	.123	.045	.058	.015	.241	2.56
5	Dissolved rock, fish, and potash.....	.170	.043	.055	.020	.288	1.53
6	Dissolved animal bone.....	.135	.053	.033	.023	.244	2.65
7do.....	.088	.038	.048	.025	.199	1.11
8	Animal bone and potash.....	.100	.063	.040	.023	.226	3.35
9	Bone tankage.....	.213	.095	.058	.030	.396	6.19
10	Complete fertilizer.....	.138	.070	.060	.018	.286	2.47
11do.....	.098	.053	.035	.018	.204	4.42
12do.....	.250	.078	.063	.043	.434	2.30
13	Complete raw bone base.....	.180	.055	.025	.045	.305	4.80
14do.....	.168	.068	.048	.055	.339	6.52
15do.....	.125	.070	.050	.025	.270	5.34
	Average.....	.129	.053	.043	.026	.251

From the results it appears that an average of 0.251 per cent of phosphoric acid was removed from the samples by the four washings of 100 cc each, over half of which (0.129 per cent) was removed by the second 100 cc of wash water. This shows that practically all the dissolved phosphoric acid was removed in the first 200 cc of wash.

A set of samples was weighed, soluble extracted as usual, treated with ammonium citrate (1.09 sp. gr.) for half an hour at 65° C., filtered, and the residues washed until the filtrate and washings amounted to 250 cc. The flasks were then changed and the residues further washed until 100 cc of water had run through each. The phosphoric acid was determined in these last washings, with the following results:

TABLE II.—Per cent of P_2O_5 removed by 100 cc water after washing with 150 cc H_2O .

No.	Fertilizer.	P_2O_5 removed.	No.	Fertilizer.	P_2O_5 removed.
1	Dissolved South Carolina rock....	0.030	9	Bone tankage.....	0.068
2do.....	.041	10	Complete fertilizer.....	.040
3	Dissolved South Carolina rock and potash ..	.041	11do.....	.075
4do.....	.045	12do.....	.045
5	Dissolved rock, fish, and potash ..	.045	13	Complete raw bone base ..	.063
6	Dissolved animal bone.....	.068	14do.....	.068
7do.....	.033	15do.....	.055
8	Animal bone and potash.....	.058		Average.....	.051

In no case was as much as 0.10 per cent phosphoric acid removed by this extra washing. It will be noticed, however, that the amount is much greater in those containing animal matter.

While the amounts removed even by as prolonged washing as in the first experiment are small, yet differences in the amount of washing the insoluble residue by different analysts may account partly for the differences in results on insolubles. In view of this possibility it might be advisable for the association to set the amount that the insoluble residues shall be washed.

Mr. WILEY. I have a paper here from Mr. Gladding, who has requested me to bring it before the association.

DISCUSSION ON THE ESTIMATION OF PHOSPHORIC ACID.

By T. S. GLADDING.

The following work may prove of interest to the members while considering the subject of the Pemberton-Kilgore method of estimating phosphoric acid:

A standard solution of microcosmic salt, 10 grams per liter, was taken for test experiments. A series of determinations, using 25 cc of solution containing 0.250 gram salt was made by two methods. First, the gravimetric method published by myself (Journal Amer. Chem. Soc., Vol. XVII, No. 1), and second, by the volumetric or Pemberton-Kilgore process. The microcosmic salt was the same salt that was used in the work on the gravimetric estimate of P_2O_5 as phosphomolybdate of ammonia, and the results are comparable with those obtained at that time. All results are calculated to percentage results.

Series.	Gravimetric method.		Volumetric method.	
	P_2O_5 found.	Theory.	P_2O_5 found.	Theory.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1.....	34.04	34.07	34.10	34.07
2.....	34.04	34.07	33.90	34.07
3.....	34.00	34.07	34.10	34.07
4.....	34.14	34.07	34.10	34.07

A number of comparative tests of fertilizers have also been made.

Series.	P_2O_5 found.	
	Gravimetric method.	Volumetric method.
	<i>Per cent.</i>	<i>Per cent.</i>
1.....	16.68	16.60
2.....	16.90	16.94
3.....	1.22	1.19
4.....	1.11	1.11

It was also found that the yellow salt after drying at $105^\circ C$. could be titrated with good results. Although the blue color of the solution was very pronounced, yet by diluting considerably and using a strong, clear light a very sharp reaction could be obtained. The results were as follows:

Series.	P_2O_5 found.		Series.	P_2O_5 found.	
	By weighing yellow salt.	By titrating the same.		By weighing yellow salt.	By titrating the same.
	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>
1.....	16.42	16.32	8.....	15.30	15.38
2.....	10.11	10.06	9.....	11.00	11.12
3.....	16.59	16.54	10.....	0.85	0.88
4.....	3.48	3.38	11.....	19.48	19.48
5.....	9.25	9.17	12.....	30.12	30.15
6.....	8.44	8.42	13.....	30.01	30.00
7.....	15.50	15.46	14.....	3.24	3.30

These results are very accordant, and prove the uniform composition of the yellow precipitate when precipitated according to directions given in my paper. But it was found that when the molybdate solution was added all at once or suddenly to the solution an excess of precipitate, when dried and weighed, was obtained. The occluded impurity does not seem to interfere with the accuracy of the volumetric process when titrated without previous drying. Its nature has not yet been determined. For gravimetric work a gradual addition of phosphomolybdate solution is essential.

In all the results given above the filtering was done rapidly by means of a pump, and three or four washings of 20 cc each were found sufficient to remove nitric acid from the yellow precipitate. At this point a very important difficulty was experienced. On one occasion several precipitates were given to an assistant to filter and wash until the washings were no longer acid, as per instructions given in method of 1895 meeting. He reported, after some time, that the washings continued acid. An inspection revealed the fact that the precipitate was clearly dissolving in the water employed for washing.

A very thorough examination into the matter seems to show that after all the nitric acid has been removed from the yellow salt a decided solvent action is exerted upon the yellow salt, such solvent action increasing with the amount of the precipitate and with the time of exposure to the water.

Thus, after washing with the pump, as described above, a further washing with 150 cc of distilled water, using about 2 grams of yellow precipitate and taking about an hour for the filtration, dissolved 0.0013 gram P_2O_5 ; a second treatment with 250 cc water dissolved 0.0025 gram P_2O_5 . The yellow salt was now shaken with water for some hours and allowed to stand overnight. One hundred cubic centimeters of clear filtrate was found to contain 0.0063 gram P_2O_5 . The same salt was now treated in the same way with 1 per cent nitric acid solution. One hundred cubic centimeters were found to contain 0.003 gram P_2O_5 . In like manner 100 cc of 10 per cent KNO_3 solution when saturated in same way were found to contain 0.004 gram P_2O_5 . In two cases the water washings came through a perfectly clear solution, yet decidedly yellow in color, and 100 cc of washings contained 0.010 gram P_2O_5 . In this work all ammonia vapors were excluded.

I can venture no explanation of these singular experiences, so widely different from the results obtained by Kilgore and others. They show that when the yellow salt is washed too long a decomposition or solution in water seems to take place that may seriously interfere with the accuracy of the results. Either some very strict directions as to washing, limiting the time and amount, or, still better, the finding of some neutral washing liquid that will not exert this solvent action, would seem to be necessary. The trouble which I have experienced is certainly a very remarkable one and requires explanation before the method can be considered as reliable.

Mr. McDONNELL. There is a point right here I do not understand in regard to the use of nitric acid. What is the advantage of using nitric acid as a wash when we must use the same amount of water to wash out the nitric acid? That is a question which has been presented to the association before in considering the washing of this yellow precipitate with ammonium nitrate. It was shown by experiments of several members of this association, by comparing results, that 10 per cent ammonium nitrate and washing with pure water brought practically the same results. If water would reduce the amount of phosphoric acid present, certainly in the former case the same thing would take place.

Mr. WHEELER. I would like to hear from Mr. Kilgore in reply to what has been said.

Mr. KILGORE. The question, as it has occurred to me, in the work I have done upon this method and upon phosphoric acid in general, is that in our regular method we wash with water and do not lose anything, because the washing with water is not carried to a sufficient extent. I consider if the washing were carried to a sufficient extent we would lose. In the washing, after washing with water in the volumetric method, as in this case, it is not carried far enough for this solvent action to take place. As to the use of nitric acid, I feel that it is hardly necessary to say more than I have said in my reports of the last two years. The nitric acid is used to dissolve molybdic acid precipitated. The nitric acid was used to remove that, then the dilute potassium was used to remove the nitric acid, and then the continued washing with water was sufficient to remove—not necessarily to remove this potassium nitrate—any remaining nitric acid that might be there, but not carry the washing to a sufficient extent to dissolve the precipitate. Washing with 250 or 300 or sometimes 400 cc will not affect the precipitate. I consider it perfectly safe to wash with 300 cc and usually with 400, but I would not hesitate to wash with 300.

Mr. FREAR. I would like to ask a question. Have you proven to your own satisfaction that after washing with 250 cc of water you get a solution which is not acid?

Mr. KILGORE. You will get a solution which is very slightly acid. After washing with potassium nitrate and some water there will be a very slight acidity. I reported in last year's proceedings, you will notice, that the acidity was not enough to affect results. I combined the washings after washing first with nitric acid and the potassium nitrate, and I then washed a number of precipitates and made these in triplicate; used several and then evaporated them and determined the phosphoric acid, and I figured that and found it only amounted, where I used a liter of water, to 0.200 per cent. It is only very slightly acid.

Mr. FREAR. That was my general recollection of what Mr. Kilgore said, and the question came strongly to me whether it would not be wise to so modify the language of the method that so experienced a chemist as Mr. Gladding would not be misled by the language of the method. Say "until the filtrate is no longer acid." It certainly would be misleading as it is. Would it not be sufficient to say, "wash with no more than 250 cc of water?"

Mr. KILGORE. That was practically the recommendation I made last year.

Mr. FREAR. I was going to move that the consideration of this wording be referred to the committee on recommendations when they report.

Mr. WILEY. I would like to present a paper relating to phosphatic fertilizers.

THE MECHANICAL ANALYSIS OF BASIC PHOSPHATIC SLAGS.

By H. W. WILEY.

Before discussing the subject-matter of the paper which I now present I think it desirable to give a short sketch of the earlier literature relating to basic slags and the methods of their valuation.

In 1882, while examining some very finely ground bone in the laboratory at Purdue University, Lafayette, Ind., I noticed a marked solubility of the phosphoric acid therein in neutral citrate of ammonia. At that time Mr. H. A. Huston, who was a student in my laboratory, was directed to make some investigations of this phenomenon, and the results of his work were incorporated in his thesis for graduation, and the data were published in the proceedings of the agricultural college of Indiana, Purdue University, for the year 1882.

In 1886 Jensch noticed that when lime and tricalcium phosphate were ignited together and the resulting mass pulverized, both citric acid and neutral ammonium citrate would extract phosphoric acid from the powder. (Berichte, vol. 19, p. 3093.)

In 1887 I published in *Agricultural Science* (March number) an article on the use of phosphorus contained in iron ores as a fertilizing material after the conversion of the ores into iron or steel.

In 1889 I received from the Pottstown Steel Works a sample of phosphatic slag which was sent me under the name of cinder. The superior merits of basic phosphatic slags as a fertilizer led me to believe that they might be largely soluble in neutral citrate solution. I therefore had this sample examined by Messrs. McElroy and Krug with that object in view. It was found to contain 20.20 per cent total phosphoric acid, of which 12.25 per cent were soluble in neutral ammonium citrate, the official solution used for the analysis of phosphatic fertilizers. An account of this property was published in the *Journal of Analytical Chemistry*, October, 1889. In this paper it is stated: "From the above results it is seen that in the phosphate slag we have a form of phosphoric acid equivalent in value to a first-class superphosphate."

Petermann, in 1889, called attention to the fact that the calcium phosphatic limestone gives up phosphoric acid to a citrate solution. (*Chemisches Central-Blatt*, 1889, vol. 2, p. 1036.)

In 1890 Jensch proposed the use of citric acid for the separation of tetra from tricalcium phosphate.¹

In 1892 Foerster published the statement that tetracalcium phosphate is easily soluble in ammonium citrate solution.²

Wagner proposed a special ammonium citrate solution having an excess of citric acid as a solvent for the tetracalcium phosphate in basic slags, and published an account of it in 1893.³

Since then the literature on this subject is very extensive, and it would be useless to attempt to cite the papers which have been published. It appears from a study of the literature that the first published account of the application of ammonium citrate as a method of valuing the availability of phosphatic slag was made from this laboratory in the paper published in the *Journal of Analytical Chemistry* above noted. For some reason, however, the method itself is universally known as the Wagner process.

Experience has shown that the official method of determining available phosphoric acid applicable to the reverted phosphoric acid in superphosphates is of little value when applied to phosphatic slags. The composition of phosphatic slags has been previously described by Krug and myself.⁴ It is evident that the tetrabasic lime

¹ *Zeitschrift für Angewandte Chemie*, 1890, p. 504.

² *Zeitschrift für Angewandte Chemie*, 1892, No. 113.

³ *Chemiker Zeitung*, vol. 13, p. 1153.

⁴ *Jour. Anal. and Applied Chemistry*, vol. 5, p. 685.

phosphate is less stable than the tricalcium phosphate, and hence the phosphoric acid which it contains is more readily given up to the growing plant. When a basic phosphatic slag is treated by neutral ammonium citrate in the regular official manner, the quantity of phosphoric acid which passes into solution is far less than the available content of phosphoric acid as indicated by the effect of the fertilizer upon a growing crop. For this reason other methods of determining available phosphoric acid have been proposed, and the one now in use is known as the Wagner process, in which a strongly acid ammonium citrate solution is used as a solvent.¹ The method of solution employed is a purely arbitrary one, but gives results comparable to the effects produced in the field.

The action of this solvent on phosphatic slags is very different for different samples. In some cases almost the whole of the sample is composed of pure tetracalcium phosphate, and in these instances the solvent action of the reagent is very high. In other cases the sample may contain tricalcium phosphate, particles of siliceous slags, and also of iron and steel. In these instances a low solvent action is noticed. It therefore has seemed desirable to supplement the chemical analysis of the slags with a mechanical separation. For particles of one-tenth of a millimeter in diameter, and above, this separation is easily effected in a series of sieves, the finer sieves being made of bolting cloth. It is evident that a further separation of the fine particles can not be secured by water, by reason of the action of this reagent upon the free lime which the slag contains. About four years ago I made some experiments looking to the substitution of alcohol in the place of water in the mechanical analysis of soils. The preliminary experiments succeeded so well that it was deemed advisable to use alcohol for securing the separation of the finer particles of the slag. The separations were made for me by Mr. C. C. Moore, of the laboratory staff. The method of procedure is as follows:

From 20 to 50 grams of the slag are placed in a shaking bottle and vigorously agitated for a few minutes on a milk-shake machine. This treatment serves to break up all the flocculates the sample may contain. The sample is placed in beakers entirely similar to those which are used in the mechanical separation of the soils by the subsidence method. At first it was only desired to secure two series of separates of the fine particles, and these were obtained by an arbitrary time standard of subsidence. All the particles which remained in suspension in a column of 15 centimeters of alcohol for five minutes were collected together and marked as separate No. 5. When all the particles which thus remained in suspension had been removed another separation was made, consisting of those particles remaining in suspension in like conditions for two minutes and a half. This portion of the sample was collected and was designated as separate No. 4. The remaining portion of the sample was freed of alcohol and separated into three portions, by sifting through two sieves made of bolting cloth with meshes one-tenth and one-quarter of a millimeter, respectively. All that portion passing through the one-tenth millimeter mesh is designated as separate No. 3. The portion passing the quarter millimeter mesh but not passing the tenth millimeter mesh is designated as separate No. 2. The portion not passing through the quarter millimeter mesh is designated as separate No. 1. The remarkably uniform size of these separates is shown by photographs published in the *Journal of the American Chemical Society*, Vol. 19, p. 19. By an inspection of separate No. 5 it is seen that it could be easily divided, and another and finer portion, by means of a ten-minutes' subsidence in alcohol, be secured.

The alcohol used for the separation is of 90 per cent strength.

The original sample and the separates were subjected to a chemical analysis, conducted for me by Mr. E. G. Runyan, of the laboratory staff. The analytical work

¹ Wiley's *Agricultural Analysis*, vol. 2, p. 80.

was confined to a determination of the total phosphoric acid and that soluble in the Wagner reagent. The data obtained follow:

Substance.	Original sample.	Separate No. 1.	Separate No. 2.	Separate No. 3.	Separate No. 4.	Separate No. 5.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Total P_2O_5	16.54	13.91	16.14	16.71	18.21	18.16
Citrate soluble P_2O_5	11.67	6.24	10.85	12.21	13.65	14.91
Proportion of original sample		12.00	33.00	26.00	8.00	21.00

From the above analyses it is seen that the separates 4 and 5 contain not only the highest total quantity of phosphoric acid, but also the highest available acid. It is seen that there is a regular progression in the richness and availability of the sample from the coarsest to the finest parts. The relative proportions of the different separates to the whole are shown in the table. The data lead to the inference that it may be advantageous to the manufacturer of basic slags to secure at least a partial separation in some such manner as indicated, for the purpose of being able to place on the market a more concentrated and available fertilizing material. The annual product of basic phosphate slags now amounts to about 1,500,000 tons and the output is rapidly increasing. Up to the present time American analysts have not had occasion to examine many samples of phosphatic slags, but it is certain, in the near future, that the consumption of this material in the United States will rapidly increase. It is important, therefore, that investigations be made looking to the best methods of chemical and mechanical analyses.

The PRESIDENT. Are there any more papers on the subject of phosphoric acid? Is there any further discussion of the subject? If not, since the hour for adjournment is near at hand, we will now take a recess until 2 o'clock p. m.

SATURDAY—AFTERNOON SESSION.

The meeting was called to order by the president at 2 o'clock. The regular order was the report on soil and ash analysis, which was presented by Mr. Goss.

REPORT ON SOILS AND ASH.

By ARTHUR GOSS, *Reporter*, and W. G. BROWN, *Associate Reporter*.

As a preliminary step in the soil work this year, a circular letter was sent to the chemists of the different experiment stations inquiring what subjects it was thought best to take up. From the replies received, and in accordance with the views of the reporters, it was thought best to limit the chemical work on soils to a further study of methods for determining the availability of phosphoric acid and potash. As a considerable number of chemists also expressed a desire to have a study of the physical properties of soils included, upon request, Prof. Milton Whitney, Chief of the Division of Agricultural Soils, United States Department of Agriculture, kindly consented to furnish the working directions.

Much time was consumed in searching for suitable samples. In this connection, a circular letter was sent to the chemists and agriculturists of the different stations asking concerning samples of soils which had been proven deficient in available phosphoric acid and potash by a number of years' scientific field-fertilizer tests. After a considerable amount of correspondence, typical potash samples were secured, through the kindness of Mr. A. M. Peter, from the Kentucky Experiment Station farm, and a soil proven deficient in available phosphoric acid from Massachusetts,

through the kindness of Mr. William P. Brooks, of the Hatch Experiment Station. As a sample containing plenty of available phosphoric acid, one was taken from a field on the New Mexico Experiment Station farm. After having received the results of the work on these soils, the reporter feels sure that more typical samples could not well have been found for the points under consideration.

The following descriptions of the samples and working directions were sent to the chemists who had consented to cooperate:

DIRECTIONS FOR WORK ON THE A. O. A. C. SOIL AND ASH SAMPLES FOR 1896.

DESCRIPTION OF THE SAMPLES.

Soil sample No. 1 is from Acre P, plat 2, of the Kentucky Experiment Station farm. (See Kentucky bulletins.) It has been cultivated for a number of years without the addition of any fertilizer, and will not, at present, produce a large crop. From several years' fertilizer tests on adjacent plats, it has been shown that in all probability the trouble is lack of available potash.

Soil sample No. 2 is also from the Kentucky Experiment Station farm. This soil has not been under cultivation, at least for a number of years, and in all probability contains sufficient available potash to produce several good crops.

Soil sample No. 3 is from the farm of Mr. Dole, of Shelburne, Mass. The results of fertilizer tests on this soil have been reported in Bulletins 14, 18, etc., of the Hatch Experiment Station. It has usually responded to phosphatic fertilizers during several years' tests, and in all probability does not, at present, contain sufficient available phosphoric acid to produce a large crop.

Soil sample No. 4 is from the New Mexico Experiment Station farm in the Rio Grande Valley. Judging from the crops produced, it contains a good supply of available phosphoric acid. It is probably about an average soil in the amount of total phosphoric acid contained.

Sample No. 2 probably contains an unusually large amount of available phosphoric acid, and No. 4 an unusually large amount of available potash.

The ash sample is from New Mexico grown alfalfa, or lucern.

In the directions given below, unless otherwise stated, references are to the "Methods of analysis adopted by the A. O. A. C., September, 1895," published as Bulletin No. 46 of the Chemical Division, United States Department of Agriculture.

WORKING DIRECTIONS FOR THE ASH.

Analyze by the association method, page 44. The sample is ready for analysis.

WORKING DIRECTIONS FOR THE SOILS.

The samples are sifted ready for analysis. Samples 1 and 2 are to be worked for potash only, and samples 3 and 4 for phosphoric acid only, except No. 4, in which both potash and phosphoric acid are to be determined, as outlined below in No. 7. All results are to be calculated to the water-free basis.

All reagents used should be tested by running blank determinations with at least the quantities used in the regular determinations.

1. *Moisture.*

Determined by the association method, page 38.

2. *Treatment with citric acid.*

Prepare a one-fifth normal citric-acid solution by dissolving 14 grams of the crystallized acid in each liter of water used and bringing to exact strength by titrating against carbonate-free KOH, using phenolphthalein as an indicator. Transfer 100 grams of each of the samples to half gallon, salt-mouth, glass-stoppered bottles, or other similar vessels; pour on each exactly 1 liter of N/5 citric acid, place in a large water bath (a metal washtub will do), and keep at the temperature of 40° for exactly five hours, shaking at frequent intervals. At the end of the digestion shake contents of bottle well and pour quickly upon a large, dry, ribbed filter of two thicknesses of paper and of sufficient size to receive the entire contents of the bottle. The filtrate is to be received in a dry vessel and the solution poured back through the paper until it is entirely free from soil particles. Evaporate 500 cc of the filtrate, corresponding to 50 grams of soil, to a small volume in a porcelain dish, transfer to a platinum dish, bring to dryness or nearly so on the water bath, finish drying in the air bath at a higher temperature, and burn out the organic matter at as low a heat as possible, in order to avoid volatilizing potash. Transfer the residue to a small porcelain dish, cover, and add about 20 cc of concentrated HCl.

When effervescence has ceased remove the cover and evaporate to dryness on the

water bath; finish, if necessary, in the air bath at a slightly higher temperature. Take up the residue with 2 or 3 cc of strong HCl and a little water, digest on the water bath until the iron compounds have been dissolved, filter off the carbon, silica, etc., wash with hot water, and determine potash or phosphoric acid in the filtrate (Filtrate A), as follows:

(A) K_2O . To be determined in samples 1 and 2 only.

Dilute Filtrate A to about 100 cc, heat to boiling, precipitate with ammonia and ammonium oxalate in slight excess, filter, wash thoroughly with hot water, evaporate filtrate to dryness in a platinum dish, add 1 cc of 1 to 1 H_2SO_4 , and expel ammonium salts by ignition. Take up residue with water and a few drops of ammonium-free HCl, filter if necessary, add 2 cc of the usual platinum-chlorid solution, and finish the determination as under the Lindo-Gladding method, page 3.

If preferred, the potash may be determined directly in Filtrate A by the following modification of the Tatlock method, as recommended by Dyer (see Journal of the London Chemical Society, March, 1894): "To Filtrate A in a platinum dish add 2 cc of platinum-chlorid solution, evaporate to dryness, take up with 80 per cent alcohol, filter, wash with alcohol, ammonium-chlorid solution, etc., and proceed as under the Lindo-Gladding method. This method is much quicker than the usual one and has been found to give good results. It is to be observed, however, that the HCl used in dissolving the residue after ignition must be free from ammonium salts." In reporting results please state which method was used.

(B) P_2O_5 . To be determined only in samples 3 and 4.

Add ammonia to Filtrate A to alkaline reaction, then add nitric acid to acid reaction and evaporate to about 50 cc. Add 10 grams of ammonium nitrate, heat to 80° , add at least 50 cc of recently prepared molybdate solution, allow to stand in a warm place for 12 hours, filter off precipitate, wash with ammonium-nitrate solution acidulated with nitric acid, dissolve precipitate in warm ammonia and precipitate with magnesia solution in the usual manner.

If preferred, the P_2O_5 may be rapidly precipitated after neutralizing with ammonia, acidifying with nitric acid, and adding ammonium nitrate, as recommended in the proceedings of the Eleventh Annual Convention of the A. O. A. C., page 58. In this case the solution should be diluted (100 cc) and a freshly prepared molybdate solution used.

3. Treatment with ammonium citrate.

Treat 100 grams of each sample exactly as under citric acid, No. 2, with 1 liter of neutral ammonium citrate solution, containing 100 cc of the usual fertilizer citrate solution, prepared as under "Optional method," page 11.

4. Treatment with oxalic acid.

Prepare a one-fifth normal solution of oxalic acid by dissolving 12.6 grams of the crystals in each liter of water used, and bringing to exact strength by titrating against standard carbonate-free KOH, using phenolphthalein as an indicator. Treat 100 grams of each sample with 1 liter of this solution exactly as under No. 2.

5. Treatment with ammonium oxalate.

One hundred grams of each sample are to be treated with 1 liter of ammonium-oxalate solution containing 16 grams of the crystals, exactly as under citric acid.

6. Treatment with hydrochloric acid.

Digest 100 grams of each sample with 1 liter of fifth normal HCl for five hours, at 40° , as before. Filter and evaporate 500 cc of the filtrate to dryness in a porcelain dish; after adding a few cubic centimeters of nitric acid to oxidize organic matter, continue the drying in the air bath until complete dryness is reached, take up with water and a few cubic centimeters of HCl, filter and determine K_2O or P_2O_5 in the filtrate (Filtrate A), as given under No. 2.

7. Effect of varying the amount of solvent.

Treat amounts of 200 grams each of sample 4 with 1, 2, 3, 4, and 5 liters of fifth normal citric acid, and determine both K_2O and P_2O_5 in each filtrate, as under No. 2. Solutions corresponding to 50 grams of soil are to be precipitated in each case.

8. Physical examination of the soils.

For working directions for the physical examination of the soil samples, reference will be made later to a bulletin by Professor Whitney, of the Division of Agricultural Soils, United States Department of Agriculture. This bulletin is now in press.

Any other results on the above, or other soils of known agricultural properties, will be thankfully received.

In the preparation of the above directions free reference has been made to the directions of the reporter of last year.

Use Bulletin No. 4, of the Division of Agricultural Soils, United States Department of Agriculture, on Methods for the Mechanical Analysis of Soils, by Prof. Milton Whitney, for the working directions. Begin with sample No. 1 and page 9 of the bulletin and do as much as you can find time to accomplish.

TABLE NO. 1.—*Soil samples.*

Analyst.	Sample No. 1.							Sample No. 2.						
	Moisture, association method.	K ₂ O. 100 grams of soil and 1 liter of solvent used. Water-free basis.						Moisture, association method.	K ₂ O. 100 grams of soil and 1 liter of solvent used. Water-free basis.					
		N 5 HCl.	N 5 CaCl ₂ .	N 5 citric acid.	Citrate solution, diluted 1 in 10.	N/5 oxalic acid.	Am. oxalate, 16 grs. per liter.		N 5 HCl.	N 5 CaCl ₂ .	N/5 citric acid.	Citrate solution, diluted 1 in 10.	N 5 oxalic acid.	Am. oxalate, 16 grs. per liter.
John Thompson, South Carolina	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
C. C. Moore, United States Department of Agriculture	2.25	0.011	0.006	9.006	0.005	0.016	3.01	0.024	0.014	0.022	0.014	0.030
R. F. Hare, New Mexico.	1.72	.011006	.005	.011	.011	2.05	.033023	.020	.038	.016
Arthur Goss, New Mexico.....	1.71	.013008	.010	.010	.011	1.95	.022031	.022	.024	.034
Average	1.83	.012	{ ^{0.006} _{.004} }	.007	.008	.011	.009	2.10	.024	{ ^{0.015} _{.015} }	.020	.020	.022	.021
Average	1.88	.012	.005	.007	.007	.009	.012	2.28	.026	.015	.022	.021	.024	.025

TABLE NO. 2.—*Soil samples.*

Analyst.	Sample No. 3.							
	Moisture, association method.	Total P ₂ O ₅ Goss method.	P ₂ O ₅ . 100 grams of soil and 1 liter of soil used. Water-free basis.					
			N/5 HCl.	N/5 CaCl ₂ .	N/5 citric acid.	Citrate solution diluted 1 in 10.	N/5 oxalic acid.	Am. oxalate, 16 grs. per liter.
John Thompson, South Carolina...	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
C. C. Moore, United States Department of Agriculture	2.26	0.020	0.034	0.026	0.080	0.012
R. F. Hare, New Mexico.....	1.59	0.22	.019032	.018	.096	.010
Arthur Goss, New Mexico.....	1.67	.21	.019	0	.023	.019	.097	.010
Average	1.84	.22	.019	0	.033	.021	.091	.011

Analyst.	Sample No. 4.											
	Moisture, association method.	Total P ₂ O ₅ Goss method.	P ₂ O ₅ . 100 grams of soil and 1 liter of solvent used. Water-free basis.								K ₂ O.	
			N/5 HCl.	N/5 HCl corrected for neutralization.	N/5 CaCl ₂ .	N/5 citric acid.	N/5 citric acid corrected for neutralization.	Citrate solution diluted 1 in 10.	N/5 oxalic acid.	Am. oxalate, 16 grs. per liter.	N/5 HCl corrected for neutralization.	N/5 CaCl ₂ .
John Thompson, South Carolina	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
C. C. Moore, United States Department of Agriculture	5.61	0.051	0.025	10.024	0.083	0.013
R. F. Hare, New Mexico.	3.26046027004	.076	.009
Arthur Goss, New Mexico.....	4.26	0.15	.049025005	.086	.008
Average	4.65	.13	.051	0.081	0	.024	0.033	.004	.088	.009	0.090	0.051
Average	4.45	.14	.049	.081	0	.025	.033	.004	.083	.010	.090	.051

¹ Not included in the average.

TABLE No. 3.—*Soil samples.*

Analyst.	Sample No. 4. Different amounts of same solvent. 200 grams soil.									
	K ₂ O. Water-free basis.					P ₂ O ₅ . Water-free basis.				
	1 liter N/5 citric.	2 liters N/5 citric.	3 liters N/5 citric.	4 liters N/5 citric.	5 liters N/5 citric.	1 liter N/5 citric.	2 liters N/5 citric.	3 liters N/5 citric.	4 liters N/5 citric.	5 liters N/5 citric.
John Thompson, South Carolina.....	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
	0.020	0.041	0.058	0.069	0.004	0.028	0.031	0.035
C. C. Moore, United States Department of Agriculture.	.032	.055	.069	.077	0.106	.004	.023	.030	.035	0.046
R. F. Hare, New Mexico.....	.025	.051	.066	.074	.085	.004	.027	.028	.036	.041
Average026	.049	.064	.073	.095	.004	.026	.030	.035	.043

TABLE No. 4.—*Alfalfa ash.*

Substance.	By C. C. Moore, U. S. De- partment of Agricul- ture.	By A. M. Holt, New Mexico.	Substance.	By C. C. Moore, U. S. De- partment of Agricul- ture.	By A. M. Holt, New Mexico.
	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>
Soluble SiO ₂	3.76	3.72	SO ₃	4.79	4.53
Insoluble SiO ₂ (sand).....	6.23	5.72	CO ₂	18.10	17.94
Total SiO ₂	9.99	9.44	Cl	3.80	4.21
K ₂ O.....	21.20	21.27	Carbon.....	9.54	10.34
Na ₂ O.....	2.95	2.83	Moisture.....	.61	.76
CaO.....	20.45	20.32	Total.....	100.87
MgO.....	3.34	3.39	O equivalent of Cl.....	.86
Mn ₃ O ₄12	.05	Corrected total.....	100.01
Fe ₂ O ₃ , Al ₂ O ₃	2.71			
P ₂ O ₅	3.28	3.27			

THE RESULTS.

It would seem that the soil reporter has, as usual, been guilty of asking for a larger amount of work than many men so busy as the chemists of the agricultural experiment stations could well find time to undertake. It is probably largely for this reason that no results have been received on the physical investigation of the soils and but few on the chemical soil and ash work.

The results of the ash work are given in table 4. While the reporter has been aware that the method of analysis for the ash is badly in need of revision in some respects, owing to lack of time nothing has been attempted in that connection this year.

The results on the soils are given in tables 1, 2, and 3. While few in number, some very interesting points are brought out. The reporter feels not a little encouraged in believing that we may at last have methods at hand by which it may be possible to tell at least the difference between a soil containing little available potash or phosphoric acid and one containing a considerable amount of those substances. Much more work will of course be necessary, however, before we can form any very definite conclusion in regard to these methods.

The agreement of results this year is quite as good as could be expected, and

the reporter wishes to take this opportunity to thank those who have cooperated in the work.

Of the solvents tried the organic acids may, I think, after two years' trial by the association, be dismissed at once as unsuited for the determination of available phosphoric acid and potash in soils, and especially so far as the P_2O_5 is concerned. While all the solvents have shown a marked difference between the two potash soils, and that in accordance with the evidence furnished by the field tests, the organic acids are no better in that respect than hydrochloric acid, and especially calcium chlorid, and besides, owing to the large amount of organic matter to be burned out, are much more difficult to work, and consequently more liable to yield discordant results.

In the case of phosphoric acid, the organic acids and salts have given utterly conflicting and misleading results. Take the case of the diluted fertilizer citrate solution, for example. In the case of soil No. 4, which experience in the field has proven contains a sufficient supply of available phosphoric acid, 0.004 per cent is secured as the average of three closely agreeing results, while in the case of soil No. 3, which has been proven deficient in available phosphoric acid, more than five times as much is gotten. The soil reporter would be very much obliged if the fertilizer chemists would kindly explain this point.

In the case of citric acid, which has also been largely recommended for this purpose, the results are also the opposite of experience in the field, and oxalic acid is little or no better.

In the case of N/5 hydrochloric acid, however, we have a solvent which, in the case of these soils, gave closely agreeing results that were entirely in accordance with the field tests. This solvent, furthermore, is very much easier to work with than the organic acids.

The two phosphoric acid soils well illustrate one point that has been overlooked by some of the investigators in soil analysis, though mentioned by a few, and that is the necessity of correcting for the neutralization of the solvent by carbonate of lime and other alkaline material when an acid solvent is used. No. 4 contains a large amount of lime (about 4 per cent), while No. 3 contains very little of that element. Hence, in the case of acid solvents, before the digestion is fairly started, No. 4 is being acted upon by a very weak solvent, whereas in the case of No. 3 it is practically full strength.

In the following table is given the dilution of hydrochloric and citric acids, in the case of these soils, as determined by titrating the filtrate at the end of the digestion against standard potassium hydrate solution, using phenol as an indicator:

TABLE 5.—*Showing neutralization of the solvent, due to the presence of lime carbonate and other alkaline material in the soil.*

Reagent.	HCl used as a solvent.				Citric acid used as a solvent.			
	1	2	3	4	1	2	3	4
Strength of acid at beginning in terms of normal acid	0.200	0.200	0.200	0.200	0.200	0.200	0.200	0.200
Strength of acid at end of 5 hours' digestion in terms of normal acid.	.191	.182	.197	.050	.175	.170	.195	.062
Per cent of solvent neutralized	4.5	9	1.5	75	12.5	15	2.5	69

By referring to table 5 it will be noticed that in the case of No. 4, 75 per cent of the acid were neutralized when using HCl, while in the case of No. 3 but 1.5 per cent were neutralized, and in the case of citric acid the difference is almost as great. The difference in the case of the two potash soils is much less.

The results secured in the case of hydrochloric and citric acids, after making the

acid of sufficient strength to allow for this neutralization, are shown in table 2. While the citric dissolved considerably more from soil No. 4 after being corrected, the results are still in no way comparable with the field tests. In the case of HCl, however, we have a solvent which, after correction, dissolved more than four times as much phosphoric acid from No. 4 as from No. 3, thus agreeing exactly with the field experience, notwithstanding the fact that No. 3 contained nearly twice as much total as No. 4. The fact that in the case of these two soils the one deficient in available phosphoric acid contains more total than the one with a good supply of available again illustrates the fact that, as is already well known to anyone who has done any considerable amount of soil work, the determination of the total phosphoric acid alone may give very misleading results concerning the amount of that substance available. The determination of the total, however, when taken in connection with the amount dissolved by dilute HCl, would seem in the case of these two soils, at least, to furnish unmistakable evidence of the more available condition of the phosphoric acid in No. 4.

It has been urged by some that a strong mineral acid, such as HCl, should not be used as a solvent for available plant food, on account of being more energetic in its action than the acids at the command of plants. As has previously been pointed out by myself and others, however, this objection will not hold in the case of at least one of the organic acids. By referring to table 2 it will be seen that oxalic acid dissolved several times as much phosphoric acid from both the phosphoric acid soils as hydrochloric acid of exactly the same strength. Oxalic acid seems to have a very strong solvent effect upon the iron compounds in the soil, and it has been my experience that the phosphoric acid usually comes into solution in connection with the iron.

Upon observing the decided increase in amount of phosphoric acid dissolved from No. 4, after correcting for the neutralization of the solvent, in the case of HCl, I was led to think that a part of the increase might be due to the solvent effect of the calcium chlorid formed. Upon trying a N/5 solution of that substance, however, it was found to have practically no solvent effect for the phosphoric acid, thus eliminating what might have been a very perplexing factor in the case. This solvent also took into solution very little of either iron, silica, or organic matter.

From the above it would seem perfectly evident that in the case of phosphoric acid, where HCl is used as the solvent, especially in the case of soils like Nos. 3 and 4, differing largely in lime content, a correction should be made for the neutralization of the acid.

While calcium chlorid has practically no solvent effect for phosphoric acid, it has a decided solvent effect for potash, as will be seen by examining Table No. 1. I consider this substance to be the most promising solvent for available potash that I have ever tried. By bringing into solution very little iron, silica, or humus the analysis is not complicated by the presence of large amounts of those substances as is usually the case with other solvents. The solvent effect of calcium chlorid is probably due to the well-known trade of bases in soil absorption, thus relieving the potash in a state of loose combination, or the presumably available, and not disturbing that locked up with the silicates. It will be observed, by referring to table 1, that calcium chlorid dissolved three times as much potash from the soil containing a good supply of available potash as from the soil known to be deficient in that substance. It is also interesting to note that in the case of soil No. 4, which is known to contain a large amount of available potash, calcium chlorid dissolved more than twelve times as much as in the case of soil No. 1.

It seems quite certain that such conditions as time and temperature of solution and strength and amount of solvent to be used must be definitely fixed if concordant results are to be expected. The plan of digesting a certain amount of soil for a week, more or less, at any temperature that may happen to prevail in the laboratory at the time, as has been practiced by some soil analysts, will not answer. Table No. 3 shows very conclusively the necessity of definitely fixing the relation between

the amount of soil and solvent. So long as this proportion remains the same, however, the results will be the same whether large or small quantities be used. This is shown in Table No. 2 by referring to the per cent of phosphoric acid dissolved in No. 4 by treating 100 grams with 1 liter of citric acid, as compared with the results in Table No. 3 from treating 200 grams of the same soil with 2 liters of the same solvent. In several trials by myself with different solvents the same result has also been reached.

METHODS OF ANALYSIS.

The reporter believes that the principal end to be sought in the agricultural chemical analysis of a soil is to secure data by which to decide, if possible, in regard to its plant-producing power. Now, it has been abundantly demonstrated that most soils contain an abundance of all the elements of plant food except phosphoric acid, potash, and nitrogen. It is on this assumption that our present system of soil fertilization rests. That this assumption is usually correct is proven by the large amount of money invested in the fertilizer industry and the unmistakably advantageous results constantly obtained by the use of fertilizers. Now, it seems to me that our practical analysis of the soil should be along these lines, especially in the East. If we can tell with reasonable certainty the amount and availability of the phosphoric acid, potash, and nitrogen in soils we have accomplished the principal thing that is at present demanded of the soil analyst.

It is interesting, to be sure, from a scientific point of view, to know the amount of iron, manganese, silica, etc., in a soil, and these things doubtless have some bearing on the question of plant production; but, owing to the time required to make such determinations and the uncertainty of the indications furnished, I do not believe that in practical work we can afford to spend time with them, at least not until we have more definite knowledge concerning the determinations of the three essential elements.

It seems to me that the thing to be done at present is to attempt to work up good, reliable methods for the determination of total phosphoric acid, total nitrogen, and either total potash or that dissolved in some strong solvent which shall give us a definite stopping point; and besides this, methods for determining the amounts of these substances that are soluble in weak solvents which shall, when taken in connection with the determinations of the totals, furnish reliable indications concerning the availability of those substances.

In the sulphuric acid method for phosphoric acid recently adopted by the association I believe we have a thoroughly reliable method for the determination of total phosphoric acid, and in the modified Kjeldahl method I believe we have an equally reliable method for total nitrogen. I do not believe that we have at present any method equally reliable and of sufficient brevity for practical purposes for the determination of potash soluble in a strong solvent; at least the discordant results reported to the association in the past have indicated this. The sulphuric acid method tried last year, however, gave results fully as good as by the other methods tried, and has the advantage of being a direct method and much quicker than the others.

In the hydrochloric acid and calcium chlorid methods outlined below we have methods for available phosphoric acid and potash which have worked well, and given results strictly in accordance with the field tests with this year's samples, and which I think should certainly be further tried.

For available nitrogen the association has at present no method, although Dr. Hilgard has recently proposed one for that purpose which I think should be tested.

RECOMMENDATIONS.

In accordance with the foregoing the reporter recommends—

1. That the complete analysis of a strong acid solution of the soil as given last year and in preceding years be stricken out of the official methods of the association.

2. That the N/5 HCl method for available phosphoric acid, as given below, be further tried by the association.

3. That the calcium chlorid method given below be further tested by the association as a method for available potash.

4. That the Kjeldahl method, as revised by the reporter, be adopted as the official method for total nitrogen.

The reporter fully realizes that the recommendations offered above are sweeping in character and involve a change in the entire system of soil analysis, but he believes that past experience and present needs fully justify such a change. We can certainly do no worse than at present, and I believe can, by placing soil analysis on a more practical basis, do very much better.

AVAILABLE PHOSPHORIC ACID.

1. Preliminary treatment to determine the neutralization of the solvent.

Weigh out 20 grams of soil, transfer to a dry flask of 400 or 500 cc capacity, add 200 cc of exactly fifth normal HCl, stop flask with a perforated rubber stopper carrying a glass tube about 2 feet long, and digest in a water bath for exactly five hours at exactly 40° C., shaking contents at frequent intervals. At the end of the digestion, filter the solution and titrate against a standard carbonate-free KOH solution, using phenolphthalein as an indicator.

2. Weigh out 100 grams of soil, transfer to a dry half-gallon salt mouth glass-stoppered bottle or other similar vessel and add exactly 1 liter of HCl of fifth normal strength plus the amount of acid neutralized by 100 grams of the soil as determined by the preliminary treatment. Immerse bottle at least to the top of the solution in a large water bath (a metal washtub is excellent), kept at a temperature of exactly 40° C., and digest for exactly five hours, shaking at frequent intervals. Remove bottle from bath, shake up contents and pour on a large dry-ribbed filter of two thicknesses of paper. The paper and funnel should be large enough to receive the entire solution; an 8-inch funnel, with paper to match, is about right. The first portion of the filtrate is to be used to rinse out the receiving vessel if the same or the funnel be wet. The solution must be poured back through the paper until entirely free from soil particles. Transfer 500 cc of the filtrate to a porcelain dish, add 2 or 3 cc of nitric acid, and evaporate to dryness, first over the lamp and finally over the water bath, moisten the residue with HCl, add hot water and digest over the water bath until all iron compounds are dissolved. Filter off the silica, receiving the filtrate in a flask of 400 or 500 cc capacity, wash with hot water, dilute the filtrate to about 100 cc if not already to that volume, add ammonia until a permanent precipitate forms, then nitric acid until the precipitate is dissolved, add 20 cc of ammonium nitrate solution containing 15 grams of the salt, heat to 80°, precipitate with 75 cc of recently prepared molybdate solution, and continue the determination exactly as under "Total acid soluble phosphoric acid," Goss method. (See methods of analysis adopted by the A. O. A. C. November, 1896.)

Reagents.—The HCl solvent is prepared by titrating against standard carbonate-free KOH, using phenolphthalein as an indicator. Phenol should be used as the indicator, as it seems to give the best results when titrating the solution after making the preliminary digestion, and it is best to use the same indicator in both cases.

The other reagents are the same as under "Total acid soluble phosphoric acid."

AVAILABLE POTASH.

Treat 100 grams of soil with exactly 1 liter of N/5 calcium-chlorid solution, exactly as under "Available phosphoric acid." Transfer 500 cc of the filtrate to a porcelain dish, add 4 or 5 cc of concentrated HCl and 1 cc of concentrated HNO₃, evaporate to dryness, take up with a small amount of water, filter, heat filtrate to boiling; add ammonia to alkaline reaction and precipitate lime, etc., with an excess of ammonium carbonate and ammonium oxalate, filter through a 12½-cm paper until clear, wash with hot water, test filtrate with a drop of ammonium carbonate to be sure that the

precipitation is complete; add .5 cc of concentrated sulphuric acid, evaporate to dryness in a large platinum dish, heat in the air bath at 150° for one hour, or until danger of popping upon ignition is past, ignite off the ammonium salts, finishing the ignition for a minute over the blast lamp. dissolve the sulphate of potash in water with the addition of 10 drops of concentrated HCl free from ammonium salts by digesting over the water bath for fifteen minutes, filter into a platinum or porcelain dish, wash with hot water, add 2 cc of platinum solution and evaporate to dryness over the water bath; add 1 cc of water and 20 cc of 95 per cent alcohol, place dish over the water bath until the alcohol begins to boil, filter, wash with alcohol, then with ammonium chlorid solution, and finally with alcohol. Good results can not be secured unless the washings are very thorough. Transfer paper and precipitate to an air bath at 110° , dry for thirty minutes, place paper in the funnel, dissolve the precipitate in water almost at the boiling temperature, receive the filtrate in a weighed platinum dish, evaporate to dryness over the water bath, dry at 110° for thirty minutes, cool in a desiccator and weigh.

Reagents.—The calcium-chlorid solution is prepared by dissolving about 12.5 grams of fused c. p. CaCl_2 in each liter of water used, filtering, if necessary, and bringing to exact N/5 strength by titrating against standard AgNO_3 solution, using potassium chromate as an indicator.

The ammonium carbonate is prepared by dissolving 250 grams of the salt in each liter of water used and adding 100 cc of strong ammonia.

The other reagents are the same as used in the official fertilizer work.

METHODS FOR THE ANALYSIS OF SOILS.

For additional methods of soil analysis, see *Methods of Analysis Adopted by the A. O. A. C.*, September, 1895, pages 38 to 43.

11.—*Total acid-soluble phosphoric acid—Goss method.*

In the case of ordinary soils, transfer 10 grams of the sifted air-dried sample, to a pear-shaped, straight-necked, hard-glass digesting flask which has been marked to hold 250 cc, and add approximately 0.7 gram of yellow oxid of mercury and 20 to 30 cc of concentrated sulphuric acid, as in the determination of nitrogen. Twenty cubic centimeters of acid are usually sufficient, but in the case of very finely divided clay soils, containing little or no sand, it is necessary to use 30 cc to prevent caking of contents of flask. In doubtful cases 20 cc of acid should first be added, and at the end of five or ten minutes, if contents show a tendency to cake, 10 cc more should be introduced. Thoroughly mix contents of flask by shaking, boil for one hour, cool (cold water can not safely be used), add about 100 cc of water, 5 cc of concentrated HCl, and 2 cc of concentrated HNO_3 , heat to boiling to oxidize iron, cool (cold water can safely be used), make up to volume and filter through a dry, folded paper until perfectly clear. (In order to secure a clear filtrate it will usually be found necessary to pour the first portion of the filtrate back through the paper three or four times.) Transfer 100 cc of the filtrate to a flask of about 500 cc capacity, add strong ammonia until a permanent precipitate forms, then 6 or 8 cc of nitric acid to dissolve the precipitate, and boil until clear. Cool the solution to 80° , add 75 cc of recently prepared molybdate solution, place the unstoppered flask for fifteen minutes in an open water bath kept at 80° C., shake flask vigorously four or five times while in the bath, then remove and let stand ten minutes to allow the precipitate to settle. Filter through a 9 cm No. 589 S. & S. paper, avoiding too strong a pressure at first, wash the flask and precipitate thoroughly with 10 per cent ammonium-nitrate solution acidified with nitric acid, place the flask in which the precipitation was made under the funnel, shut off pump and close all valves to filtering jar, fill paper half full of hot water, add a few cubic centimeters of strong ammonia, and aid solution if necessary by stirring the precipitate with a glass rod. As soon as the yellow precipitate has all dissolved open valve to the filtering jar, but do not turn on the pump at first as there is danger of loss from spattering. After the solution has

passed through, rinse paper once with a small amount of hot water, remove the flask, and place a small lipped beaker under the funnel, heat the solution in flask to boiling, and again pour through the paper, avoiding the use of the pump at first. It is well to have the beaker covered with a perforated watch glass, through which the funnel passes, to prevent loss from spattering. The flask and paper are to be washed out with a small amount of hot water (the total filtrate should not exceed 50 cc).

As has been pointed out by Hilgard, aluminum is sometimes carried down with the phosphoric acid from soil solutions when precipitating with molybdate solution, in which case some of the phosphoric acid will not be dissolved in the treatment with ammonia. This will be indicated, first, by the appearance of a white precipitate upon dissolving the yellow precipitate in ammonia; and second, by the difficulty experienced in washing. If such a precipitate be present in any appreciable quantity, proceed as follows: After washing out all the ammoniacal solution as directed above, place a small beaker under the funnel, close all valves, fill the filter one-third full of hot water, add a few cubic centimeters of concentrated HNO_3 , proceed as if dissolving phosphomolybdate in ammonia, receiving final solution and washings in the flask previously used, dilute with water to about 100 cc, add 20 cc of 75 per cent ammonium-nitrate solution, precipitate with 50 cc of molybdate solution in exactly the same manner as before, filter off the precipitate, wash, dissolve in ammonia, and add to first portion.

To the warm ammoniacal solution in the beaker, add HCl until the yellow color appears, then add a few drops of ammonia until the solution clears, cool, add 5 cc of filtered magnesia mixture from a burette, a drop at a time, with constant stirring, let stand fifteen minutes, add 20 cc of strong ammonia sp. gr. 0.90, allow to stand over night, filter, wash precipitate with dilute ammonia, dry, ignite intensely over blast lamp until white or nearly so, cool in a desiccator and weigh the $\text{Mg}_2\text{P}_2\text{O}_7$ secured.

The reagents used which have not already been described are the same as used in the official fertilizer work.

For a detailed discussion of this method see A. O. A. C. Report for 1894, page 58.

12.—Total nitrogen.

Weigh out from 7 to 14 grams of soil, transfer to an ordinary small-sized Kjeldahl digesting flask, and boil for one hour with 30 cc, or as much as may be required, of concentrated sulphuric acid and 0.7 gram of yellow oxid of mercury. Remove from over flame, and while still hot carefully add crystals of potassium permanganate until the green color is permanent after stirring, cool, fill flask about half full of water, shake contents, and after allowing the heavy portion to subside, pour the solution into a flask of 1,000 or 1,200 cc capacity, continue the washing by decantation until all the ammonium sulphate has been removed, and continue the determination exactly as under the Kjeldahl method for fertilizers, as given in the Methods of Analysis adopted by the A. O. A. C., September, 1895, pages 16 and 17.

While unnecessary in all ordinary cases, if the soil be known to contain an unusually large amount of nitrates the Kjeldahl method, as modified to include nitrates, should be used.

It is necessary to use an unusually large-sized distillation flask in order to prevent violent bumping and consequent breaking of flasks on account of the bulky precipitate present.

AMENDED AND ADOPTED REPORT OF THE COMMITTEE ON RECOMMENDATIONS IN RELATION TO SOILS.

It is recommended—

(1) That the further trial of fifth normal HCl for determining the phosphoric acid requirements of soils be referred to the reporter for 1897.

(2) That the trial of ammonium chlorid, calcium chlorid, etc., for determining the potash requirements of soils be referred to the reporter for 1897.

(3) That on page 42, section 8, under "Determination of total nitrogen," the description of the method as given by the reporter be substituted for that as printed in the proceedings for 1895.

(4) That the reporter for 1897 test the applicability of the determination of the nitrogen in the humus as a means of ascertaining the need of nitrogen fertilization (proposed by Hilgard), and that the humus for this purpose be obtained by the Huston method.

(5) That the question of the necessity for more general acid tests of soils be referred to the reporter for 1897.

H. J. WHEELER,

For Committee on Recommendations of Reporters.

The PRESIDENT. I notice that the Assistant Secretary of Agriculture is present, and I am sure we will all be glad to hear from him. He was actively connected with the association at its beginning.

Mr. DABNEY. I do not feel that I am a stranger here, although I have been away so long that I feel somewhat like a prodigal son returning to his father. I have watched with much interest the work of this association, and I regard it as the best technical association in this country, if not in the world. It has been to me a matter of pride to see the great and helpful work which it has done. I regret that circumstances over which I had no control have kept me away from the meetings of this association for so many years. I recall with pleasure the memories of my earlier associations with this body, and I would advise young men to stick to their desks, and not wander off into executive work. Molecules and atoms are far more attractive, as a rule, than men and affairs.

I thank you, gentleman, for the invitation which was extended to me to be present, and for this opportunity to say a word to you.

The PRESIDENT. The next thing in order is the presentation of papers on soil and soil analyses.

Mr. Huston read a paper on the subject.

SOIL SOLVENTS FOR AVAILABLE POTASH AND PHOSPHORIC ACID.

By H. A. HUSTON and J. M. BARTLETT.

It seems to be accepted that in the case of worn soils solution in strong mineral acid gives little insight into the availability of their potash and phosphoric acid. More recently the use of dilute organic acids, such as the 1 per cent citric acid used by Dr. Bernard Dyer¹ and the acid ammonium oxalate used by Dr. A. M. Peter,² has been tried with more promising results. The theory of the use of dilute organic acid solutions seems to rest on the idea that plant roots give off fluids containing organic acids which act on the soil in a degree comparable with the effect of the dilute acids employed in the laboratory experiments.

While I do not question that plant roots in contact with polished marble or even granite may make appreciable markings on the carbonate of lime and on the feldspar of the granite, the conditions of the experiment as usually conducted differ radically from those found in the field, for in the experiment the plants are not supplied with

¹ Jour. Chem. Soc. London, March, 1894. This paper contains a résumé of suggestions by various authors and special investigations by Dr. Dyer.

² Chem. Div. U. S. Dept. Agr., Bull. 47, p. 32.

normal soil water. So far as I have observed, normal soil waters give an alkaline reaction. No inconsiderable part of the food of the plants comes to it dissolved in the soil waters. The work of Dr. J. H. Wheeler¹ shows what marked difficulty is encountered in growing plants on a well-drained soil having an acid reaction.

Soil waters rising from a subsoil are charged with more or less of mineral salts; and if the upper layers of the soil have a different composition from the lower layers in which the soil waters have been charged we may expect chemical changes to take place according to the well established facts of soil absorption.

In view of these considerations some work was undertaken with alkaline solvents. The first solution used contained the same amount of ammonium oxalate as the solution used by Dr. Peter; but instead of the acid an amount of ammonia equivalent to the acid was added. All work is based on the same relative quantities of soil and solution as used by Dr. Peter—200 grams soil and 1,000 cc solution. In working with Dyer's solution the digestions continued at room temperature for seven days with shaking at frequent intervals. All the other digestions were continued for five hours with constant shaking in the apparatus described in Indiana Agricultural Experiment Station Bulletin 55, and Wiley's Principles and Practice of Agricultural Analysis, Volume II, page 142. The flasks were inverted every thirty seconds. The utmost care was used to secure clean precipitates of potassium platinic chlorid.

THE SOILS USED.

The Kentucky soils are those used for work by the Association of Official Agricultural Chemists for the past two years, and are described on page 31 of Bulletin 47, Chemical Division United States Department of Agriculture. Briefly stated, the soil requires the addition of potash to produce satisfactory crops of corn, potatoes, and tobacco, but seems to contain enough available potash for a good wheat crop. The field tests indicate abundance of available phosphoric acid. Soil No. 1 has received phosphoric acid and nitrogen, and soil No. 2 has received potash and nitrogen. Of the Indiana soils the one marked "Turley" is from Orange County. It is a medium clay, resting on a red clay, which in turn rests on the limestone rock of the region.

The land has been under cultivation for some seventy years and at one time was so badly worn as to be considered of very little value. The sample was drawn after plowing for corn in the spring of 1896. In 1895 wheat had been so poor on this land that hogs were turned in to eat the standing crop. In the spring of 1896 the clover was so uneven that the land was put in corn, of which it produced in this very favorable year for corn 37 bushels per acre on the unfertilized plats. The owner does not believe that it can produce a profitable crop of wheat without the use of some commercial fertilizer or manure. The usual application has been 100 pounds ground bone per acre. The field tests this year showed marked gain in corn from the use of acid phosphate and potash, but increasing the amount of potash from 30 to 60 pounds per acre gave no increased yield. Original timber, oak.

The soil marked "Campbell" is from Monroe County, and represents a cold, badly drained clay, resembling the so-called "crawfish" clay. Commercial fertilizers are considered necessary for wheat. Field tests this year showed marked gains on corn from the use of acid phosphate and potash, but increasing the amount of potash from 30 to 60 pounds per acre gave no increased yield. Original timber, poplar and mixed hard woods.

The station land is a second-bottom soil resting on gravel. It is a dark, productive loam. In favorable seasons the land will produce 50 bushels of corn and 30 bushels of wheat per acre without the use of fertilizers or manure. While commercial fertilizers have some effect in increasing the crops, the use of them on this land has not been profitable. Original timber: Black walnut, oak, maple, wild cherry, and some

¹ Rhode Island Agr. Exp. Sta., 7th Ann. Rpt., p. 152.

hickory. The plats from which the samples were drawn have been in wheat since 1888. Plats 3 E. 1 and 3 E. 4 have received no fertilizers, plat 3 E. 2 has received "complete" chemical fertilization, and plat 3 E. 5 has secured applications of barnyard manure. In five years (1890 to 1894) 3 E. 1 lost to crop 8.1 pounds phosphoric acid, 11.3 pounds potash, and 17.8 pounds nitrogen; plat 3 E. 4 lost 7.2 pounds phosphoric acid, 10.1 pounds potash, and 15.9 pounds nitrogen; plat 3 E. 2 lost net 0.8 pound phosphoric acid, 3.2 pounds potash, and 7.9 pounds nitrogen; plat 3 E. 5 gained 4.2 pounds phosphoric acid, 6.9 pounds potash, and 0.3 pound nitrogen. The plats contain one-tenth acre each. Plat 3 E. 4, a blank plat, contains humus (by Huston's method)¹ 5.3 per cent, and nitrogen in this humus 4.52 per cent. Plat 3 E. 5, which has received barnyard manure, contains humus 5.6 per cent, and nitrogen in this humus 5.71 per cent.

The mechanical analyses of the Indiana soils are shown in Table I.

TABLE I.

Source.	Gravel.	Coarse sand.	Sand.	Silt.	Clay.	Moisture and organic.	Total.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Turley	0.25	0.16	7.05	39.82	47.64	5.31	100.23
Campbell.....	1.57	.67	12.23	45.02	35.21	5.08	99.78
Station 3 E. 4.....	1.56	2.56	13.95	35.42	36.54	9.94	99.97

Since Peter's solution and the alkaline ammonium oxalate contain a salt of ammonia it was thought that the phenomena of soil absorption might come into play. To test this a solution of the same alkalinity but containing the same amount of ammonia as chlorid as was contained in the other solutions in the form of oxalate was used. To test the question of soil absorption pure and simple a neutral solution of ammonium chlorid, 17.2 grams to the liter, the same amount of ammonium chlorid as in the previous solution, was used.

The soils were also digested with ammonium hydrate, sp. gr. 0.96, containing 17.2 grams ammonium chlorid per liter, and with ammonium hydrate, sp. gr. 0.96, alone. Ammonium hydrate was tried because, as I have previously shown,² phosphates of iron and alumina are dissolved by ammonium hydrate. At first we hoped to utilize the ammonia and ammonium chlorid mixture, but in the presence of the ammonium chlorid not a trace of phosphoric acid was dissolved.

On the Kentucky soils a number of solvents were tried at a higher temperature. This modification seemed no improvement, rather the reverse, and it was decided to use room temperatures.

Table II contains the results of the work. The total potash in each soil, and the amount of potash and phosphoric acid removed by hydrochloric acid, sp. gr. 1.115, are also added for purpose of comparison.

¹Chem. Div. U. S. Dept. Agr., Bull. 38, p. 84; Wiley's Agr. Analysis, vol. 1, p. 326.

²Chem. Div. U. S. Dept. Agr., Bull. 31, p. 99.

TABLE II.

Source of sample.	Dyer's solution 100 per cent citric acid (room temp.; 7 days).	Peter's solution (room temp.; 5 hours).	Alkaline am. oxalate (room temp.; 5 hours).	Alkaline am. chlorid (room temp.; 5 hours).	Peter's solution (65° C.; 5 hours).	Alkaline am. oxalate (65° C.; 5 hours).	Alkaline am. chlorid (65° C.; 5 hours).	Ammonia, sp. gr. 0.96 (room temp.; 5 hours).	17.2 grams am. chlorid in 1 liter ammonia, sp. gr. 0.96 (room temp.; 5 hours).	17.2 grams am. chlorid in 1 liter water (room temp.; 5 hours).	Total potash by J. L. Smith method.	Soluble in HCl, sp. gr. 1.115, on steam bath.
Kentucky:	<i>P. et.</i>	<i>P. et.</i>	<i>P. et.</i>	<i>P. et.</i>	<i>P. et.</i>	<i>P. et.</i>	<i>P. et.</i>	<i>P. et.</i>	<i>P. et.</i>	<i>P. et.</i>	<i>P. et.</i>	<i>P. et.</i>
No. 3, potash and nitrogen.....	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$	(.0541) .0628	.0138 0	.0492 .0222 0	.0337 0	0	.0337 0	.0337 0	.0162 .0238	1.485	.340	.431
No. 1, phos. acid and nitrogen.....	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$	(.0560) .0776	.0165 0	.0565 .0195 0	.0352 0	0	.0352 0	.0352 0	.0148 .0137	.0218	1.467	.317
No. 1-No. 3.....	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$	(.0038) .0183	.0144 .0134	.0200 .0135	.0165 0	.0015 0	.0015 0	.0015 0	.0015 0	.0018	.023	.001
No. 3-No. 1.....	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$	(.0019) .0148	.0027 0	.0073 .0027 0	.0038 .0031	.0008 .0025	.0025 .0020	.0025 .0018	.0025 .0018	.0018	.023	.001
Indiana:												
Turley.....	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$.0028 .0031	.0025 0	.0025 .0025	.0025 .0025	.0025 .0025	.0025 .0025	.0025 .0025	.0025 .0025	.0025 .0025	.0025 .0025	.0025 .0025
Campbell.....	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$.0073 .0303	.0016 0	.0035 .0035	.0035 .0035	.0035 .0035	.0035 .0035	.0035 .0035	.0035 .0035	.0035 .0035	.0035 .0035	.0035 .0035
Experiment stations:												
3 E. 1, blank flat.....	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$.0047 .0553	.0157 0	.0304 .0304	.0304 .0304	.0304 .0304	.0304 .0304	.0304 .0304	.0304 .0304	.0304 .0304	.0304 .0304	.0304 .0304
3 E. 2, chemical fertilizers.....	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$.0267 .0244	.0150 .0176	.0073 .0177	.0331 .0331	.0331 .0331	.0331 .0331	.0331 .0331	.0331 .0331	.0331 .0331	.0331 .0331	.0331 .0331
3 E. 4, blank flat.....	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$.0053 .0530	.0156 0	.0298 .0298	.0298 .0298	.0298 .0298	.0298 .0298	.0298 .0298	.0298 .0298	.0298 .0298	.0298 .0298	.0298 .0298
3 E. 5, manure.....	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$.0025 .0447	.0140 0	.0227 .0227	.0227 .0227	.0227 .0227	.0227 .0227	.0227 .0227	.0227 .0227	.0227 .0227	.0227 .0227	.0227 .0227
	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$.0246 .0275	.0155 .0136	.0082 .0186	.0319 .0319	.0319 .0319	.0319 .0319	.0319 .0319	.0319 .0319	.0319 .0319	.0319 .0319	.0319 .0319
	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$.0019 .0479	.0157 0	.0231 .0231	.0231 .0231	.0231 .0231	.0231 .0231	.0231 .0231	.0231 .0231	.0231 .0231	.0231 .0231	.0231 .0231
	$\left\{ \begin{array}{l} P_2O_5 \\ K_2O \end{array} \right.$.0268 .0314	.0158 .0300	.0104 .0218	.0334 .0334	.0334 .0334	.0334 .0334	.0334 .0334	.0334 .0334	.0334 .0334	.0334 .0334	.0334 .0334

It will be seen that Dyer's solution and Peter's solution resemble each other in a general way in their action on the phosphates of the Kentucky soil (rich in phosphates) and on the Turley soil (poor in phosphates); while on the Campbell land (poor in phosphates) and on the station land (fairly good in phosphates) they differ radically.

In their action on potash the two solutions differ widely in the case of the Kentucky soils containing too little available potash for corn, while they resemble each other in their action on the other soils which seem from field tests with corn to contain considerably higher available potash than the Kentucky soils.

Dyer's solution extracts more phosphoric acid from the Kentucky soil that had received phosphoric acid than from the one receiving none. From the station soil it extracts the highest phosphoric acid from the soil that had received superphosphate; but it failed to extract as much phosphoric acid from the soil receiving its phosphoric acid in the form of manure as it extracted from either of the plats that had received no fertilizers. On the average, Dyer's solution extracts no more phosphoric acid from the station soils known to contain a fair supply of available phosphoric acid than from the clay soils known to be very deficient in phosphoric acid.

Dyer's solution dissolves more potash from the Kentucky soil that had received potash than from the one not receiving any.

From the clay soils, which seem from field tests with corn to be somewhat deficient in available potash, it dissolves relatively high amounts of potash. From the station soils it dissolved no more potash from the soil that had received full applications of muriate of potash than from the soils that had received no potash.

Peter's solution would indicate that there was a good supply of available phosphoric acid in the Campbell soil, where it is known to be deficient. It would also indicate that the Turley land was higher in available potash than the station soil, although the field tests indicate to the contrary.

The acid solutions of Dyer and Peter seem to fail when applied to soils of different types, although their indications are in the right direction when applied to soils of exactly the same type, such as the Kentucky soils.

The alkaline ammonium oxalate dissolves practically as much potash from the Kentucky soil as from the station soil, although the available potash is much higher in the latter, as shown by field tests.

It dissolves about the same amount of phosphoric acid from the Kentucky soil as from the station soil, both of which have a fair amount of available phosphoric acid, although the former has a much higher total phosphoric acid content. It distinguishes these soils very sharply from the clay soils known to be deficient in phosphoric acid.

The alkaline ammonium chlorid distinguishes the fertilized from the unfertilized plats very sharply on the station soil and to a fair extent on the Kentucky soil. Its action on the clay lands is in accord with what knowledge we have in regard to the potash in these soils.

The ammonium chlorid dissolved in ammonia, sp. gr. 0.96, gives results on potash in the same general direction as the mildly alkaline ammonium chlorid, but the differences are less sharply defined. As this solution is rather troublesome to work with, I would prefer to use the mildly alkaline one.

The neutral ammonium chlorid distinguishes very well the Kentucky soil from the station soil, gives fair indications on the clay soils, but fails to show the effect of the potash salts applied to the station soil.

Ammonium hydrate, sp. gr. 0.96, gives results on potash that are quite at variance with what is known about these soils, but on phosphoric acid it gives promising results. The character of the individual results indicates that the digestion was not continued long enough to complete the reaction. Yet the results clearly distinguished the lands poor in phosphoric acid from those known to be well supplied with available phosphoric acid. The only case where it seems at fault is on plat 1 of the station soil. But every other solvent acts in the same way, indicating that the sample from this plat is really higher in phosphoric acid than the sample from the other blank plat, No. 4. Crop tests covering five years show that plat 1 has a crop-producing capacity about 15 per cent greater than plat 4.

Ammonium chlorid in neutral and alkaline solution removes notable quantities of lime from soils. The quantities were determined and are given in Table III.

TABLE III.

Per cent of calcium oxid removed by various solvents.

Source of sample.	By alkaline ammonium chlorid (5 hours; room temp.).	By neutral ammonium chlorid (5 hours; room temp.).	By HCl, sp. gr. 1.115 (10 hours on steam bath).
Kentucky No. 3.....	0.144	0.173	¹ 0.51
Kentucky No. 1.....	.122	.196	¹ .31
Turley.....	.066	.122
Campbell.....	.114	.143
Station 3 E. 1.....	.096	.216
Station 3 E. 2.....	.089	.246
Station 3 E. 4.....	.113	.226	.447
Station 3 E. 5.....	.105	.227	.472

¹ Determined by Harry Snyder. See Bull. 47, U. S. Dept. Agr., Chem. Div., p. 49.

It will be seen that the alkaline ammonium chlorid removes about one-fourth and neutral ammonium chlorid about one-half as much lime as the hydrochloric acid used in the customary method of soil analysis. The station soils are practically free from carbonates, containing an average of only 0.015 per cent of carbon dioxide.

Of the solvents used we consider alkaline ammonium chlorid and neutral ammonium chlorid promising for available potash and ammonium hydrate promising for available phosphoric acid. Alkaline ammonium oxalate seems to do very well for available phosphoric acid in some cases, but the material is rather troublesome to work with on account of the large amount of organic matter that goes into solution. Where ammonium chlorid is present the solutions are nearly free from organic matter, filter very rapidly, and the ignition is readily made, leaving only a small amount of bases to be removed before determining the potash. Tatlock's method was tried for potash, but proved unsatisfactory.

We have tried chlorids of calcium, magnesium, and sodium, but prefer to use the ammonium salt, since it introduces no involatile base which would interfere with the potash determination.

We are now at work with ammonium hydrate, continuing the digestion for a longer time and changing the strength of the solution. We are also testing ammonium chlorid dissolved in 1 per cent hydrochloric acid. We shall also test the question of the relative quantities of the soil and solvent used, as the present amounts—200 grams soil to 1 liter of solvent—seems to involve too small a volume of solvent. While we feel encouraged by the outcome of the work reported above, it must be borne in mind that before any method of soil testing can be considered satisfactory, it must give reliable indications on soils of different types that have been subjected to investigation by field tests; and these tests must not be confined to one crop, but must relate to all the crops likely to be produced on the land under investigation. From data now at hand it seems probable that certain soils may have ample potash in an available form to produce good crops of cereals, while not having enough to produce profitable crops of corn, potatoes, or tobacco. This phase of the matter must be kept in mind in deciding upon any definite amount of soluble ingredients which shall be used as a minimum in judging of the fertilizer needs of a soil.

PURDUE UNIVERSITY AGRICULTURAL EXPERIMENT STATION, *November, 1896.*

The PRESIDENT. The discussion of Professor Huston's paper is in order, but the time set by the order of business for the election of officers has arrived.

We will now hear from the committee on nominations.

The committee presented the following names: For president, William Frear; for vice-president, A. L. Winton; for secretary, H. W. Wiley; for member of the executive committee, B. W. Kilgore.

The report of the committee was unanimously adopted, and the gentlemen above mentioned declared elected to the offices for which they were nominated.

The PRESIDENT. I would like to ask Mr. Wiley if it is not true that we practically have no man to become reporter on soils next year?

Mr. HUSTON. The chairman appoints the reporters.

Mr. WILEY. It would be best for this society to have the reporters serve two years instead of one, and it would not be a very great additional burden upon the reporter.

Mr. KILGORE. As I understand this matter, when it was brought up two years ago it was the intention that reporters should serve two years, and it must have been the impression of the incoming president

at that time that such was the case, because he immediately reappointed all the old reporters, and there are quite a number of them who have served two years; and I am quite sure we have a good set of reporters, and that the duty is so pleasant that, even if they do not have the time, they would be willing to work at nights to do this work.

Mr. WILEY. The constitution itself says nothing about the matter. It was really left to the discretion of the president with the recommendation that the resolution adopted at that time be carried out.

Mr. KILGORE. I think the understanding was very clear that the associate reporter was to work with the reporter and the associate was to be worked into the place, but I do not know of any case where this has been done.

So far as the real work is concerned the reporter has done it all in most every case, and I think this plan has been a pretty successful failure.

Mr. WHEELER. I think the great difficulty has been that the reporter and associate reporter have been so very far separated.

Mr. LINDSEY. Dr. Wheeler served two years and did his duty. It was my intention, when I made that motion, that the reporter should serve two years. There was a sort of entanglement. It is for the benefit of this association, as a rule, that a reporter should serve two years, and I think he ought to have the interest of the association sufficiently at heart to do it. Of course it takes a considerable portion of his time, but nevertheless it is in a good cause. A man begins as a reporter; of course he looks up the subject thoroughly and he gets started out on his work and makes out a line and accomplishes something the first year, but he learns in the first year that he has made mistakes and knows how to go to work to remedy them the next year. If you take the second year away, all the work of the first year is lost and it is transferred to another member, and he makes some mistakes and does not accomplish nearly as much for the benefit of the association as he would if he continued two years in office. With that in mind, I would very strongly favor that the reporters, for the benefit of the association, serve two years in succession.

The PRESIDENT. Do you make a motion that a provision of that kind be inserted in the constitution?

Mr. LINDSEY. How would it be to have the motion read, that "it be the sense of the association that reporters serve two years," and leave it to the judgment of the president to appoint a reporter for that period?

Mr. HUSTON. I would like to make an amendment to that motion that instead of two successive years, he be allowed to serve more than one year. I do not think we had better put a limit on him.

The PRESIDENT. Do you move that as an amendment?

Mr. BIGELOW. I would like to have the constitution read.

Constitution read.

The PRESIDENT. As many as favor the adoption of the amendment say "aye."

Amendment adopted.

The PRESIDENT. The question now is upon the adoption of the motion as amended.

Motion adopted.

The PRESIDENT. Any further paper on the subject of soils and ash analyses? If there are no further papers, the discussion of the report is in order.

Mr. KILGORE. I would suggest that it is proper to present suitable resolutions with reference to the death of a man who has been a very strong friend of this association and who has done a great deal for it—ex-Assistant Secretary of Agriculture Willits. As we all remember, the remarks before this association in the year past were very hopeful and encouraging, and I feel it is the duty of this association to appoint a committee to prepare a suitable set of resolutions. I move that a committee be appointed to draft them.

Motion carried; and the president appointed Mr. Kilgore, Mr. Huston, and Mr. Wiley on the committee.

The PRESIDENT. If there is nothing further on the subject of soils and ash, the report on dairy products is in order.

On two of the other subjects we have no formal reports, which will bring down the amount of business to a very narrow margin if we attempt to do anything more to-day. For that reason I think a motion to adjourn would be acceptable.

REPORTER ON DAIRY PRODUCTS. I should like to get rid of my report to-day. It will not take a very long time, and I shall not take more than ten minutes to dispose of it.

The PRESIDENT. What is the pleasure of the association in regard to this matter?

Moved and adopted that the reporter be allowed to read his report. Mr. Van Slyke then read his report, as follows:

REPORT ON METHODS FOR THE ANALYSIS OF DAIRY PRODUCTS.

By L. L. VAN SLYKE.

It was the purpose of the reporter to give special attention to the analysis of cheese, in order that the provisional methods might be adopted as permanent in so far as additional work seemed necessary to accomplish this result. Methods for some determinations have been so carefully worked out that they do not require any further attention.

Two points in particular have received attention: (1) Preparation of sample, and (2) determination of fat. In addition the acidity of cheese has received some study. The first of these has been found to be a matter of no small difficulty. Until this year different workers used different samples, but this year it was desired to have work done on one and the same sample.

PREPARATION OF SAMPLE.

A wedge-shaped segment was cut from the cheese, reaching from the outer edge to the center of the cheese. This was cut into strips and passed through a sausage-grinding machine three times, being mixed as completely as possible after each grinding. Small jars were filled from the cheese thus prepared and these samples sent out for analysis.

In order to ascertain how completely and uniformly mixed the samples were, determinations were made of the nitrogen in samples contained in five different jars, selected at random. The results were as follows:

Nitrogen in—				
No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
4.71	4.79	4.73	4.72	4.78
4.72	4.82	4.76	4.75	4.87

These results show that the samples of cheese sent out were as nearly uniform as it would be possible to make them, but it is possible that the fat was less uniformly distributed than the nitrogen compounds.

DETERMINATION OF FAT.

It was proposed to compare the following methods for the determination of fat:

1. The present provisional method.
2. The present provisional method modified.
3. Extraction of fat after drying.
4. Babcock test.

The present provisional method is, briefly, as follows: The sample of cheese is ground with about twice its weight of anhydrous copper sulphate and then extracted with ether.

The proposed modification avoids grinding the weighed sample until the fat has been mostly extracted, thus preventing possible loss of fat. In detail it is as follows: Use a test tube containing a perforation in the bottom, or, in place of this, an extraction thimble of fat-free paper. When a test tube is used, place at the bottom some extracted cotton, glass wool, or asbestos, and in addition wrap the lower end outside with filter paper to prevent particles of asbestos or copper sulphate being carried down through the perforation into the extraction flask. Pack the prepared tube as follows: Put first a mixture containing equal parts of anhydrous sulphate and pure incinerated sand, filling the tube for about 2 inches. Avoid packing too tightly. Upon this place a little asbestos or other suitable material. Then place the sample of cheese, which has been weighed out for analysis. It is very convenient to place the weighed sample in a little cone of copper foil or of filter paper and slip this into the top of the tube containing the copper sulphate.

The extraction is carried on as usual. After five hours the partially extracted cheese is removed and ground with pure sand in a mortar. This partially extracted cheese is brittle, and grinds up about as easily as a dry cracker. After grinding with the sand the cheese is replaced in the tube and the extraction continued ten hours longer. In the grinding there is no danger of loss of fat, because the cheese is largely extracted in five hours, and little fat remains in the residue that is ground.

The extraction after drying is accomplished as follows: Weigh out 2 to 5 grams of cheese, dry at a temperature of boiling water for ten hours, and then extract with ether. After five hours remove the cheese and grind with some incinerated sand. Replace in flask and continue extraction for ten hours longer.

The Babcock test is made in the following manner: Use cream tubes; those designed by Mr. Bartlett, of the Maine Experiment Station, are satisfactory. Test the calibration of all bottles used. Weigh the test bottle, and place in it about 8 grams of

cheese, then add about 15 cc of hot water and place upon a water bath until the cheese becomes soft, so that by shaking the bottle a uniform mixture is obtained. Complete the test in the same manner as with milk. Multiply the final reading by 18 and divide the product by the weight of cheese taken, in grams, to obtain the per cent of fat. If a sample of cheese do not dissolve readily in this way, add a few drops of strong ammonia in the bottle after the water is added, and this will soon soften the cheese so that the test can be made. Special attention is called to the precaution that the sulphuric acid should be added slowly in small portions, shaking after each addition, using a little more than is necessary for complete solution.

It is suggested that the extracted residue of cheese in each case be transferred to an ordinary Babcock testing bottle and the amount of fat in such residue determined.

While several assurances in regard to doing the work were received and samples were sent to twelve chemists, only three reported and the results in one case were much too low, owing probably to incomplete extraction.

I will present the results of work done by the following persons: S. M. Babcock, Madison, Wis.; E. N. Eaton, chemist of the Minnesota dairy and food commission, and C. G. Jenter, an assistant chemist in the New York (Geneva) Station laboratory.

Per cent of fat in cheese obtained by the provisional method.

S. M. Babcock.	E. N. Eaton.	C. G. Jenter.
(1) 38.00	(1) 38.48	(1) 38.07
(2) 38.19	(2) 38.68	(2) 38.31
	(3) 38.87	(3) 38.37
		(4) 38.41
		(5) 38.61
Average... 38.10	38.68	38.35
Lowest result.....		38.00
Highest result.....		38.87
Extreme difference87

Extreme difference expressed in per cent of total average amount of fat found equals 2.25 per cent.

Per cent of fat in cheese obtained by the modified provisional method.

S. M. Babcock.	E. N. Eaton.	C. G. Jenter.
(1) 39.33	(1) 39.60	(1) 38.72
(2) 38.85	(2) 39.73	(2) 38.85
	(3) 39.85	(3) 39.00
		(4) 39.06
		(5) 39.29
Average... 39.09	39.73	38.98
Lowest result.....		38.72
Highest result.....		39.85
Extreme difference		1.13

Extreme difference expressed in per cent of total average amount of fat found equals 2.88 per cent.

Per cent of fat in cheese obtained by extraction after drying cheese.

S. M. Babcock.	E. N. Eaton.	C. G. Jenter.
(1) 39.37	(1) 40.00	(1) 39.06
(2) 39.57	(2) 40.13	(2) 39.10
	(3) 40.26	(3) 39.75
		(4) 39.94
Average... 39.47	40.13	39.46
Lowest result.....		39.06
Highest result.....		40.26
Extreme difference		1.20

Extreme difference expressed in per cent of total average amount of fat found equals 3.02 per cent.

Per cent of fat in cheese obtained by the Babcock test.

S. M. Babcock.	E. N. Eaton.	C. G. Jenter.
(1) 39.29	(1) 39.83	(1) 39.74
(2) 39.29	(2) 39.94	(2) 39.98
	(3) 40.05	(3) 40.29
Average... 39.29	39.94	40.00
Lowest result.....		39.29
Highest result.....		40.29
Extreme difference		1.00

Extreme difference expressed in per cent of total average amount of fat found equals 2.40 per cent.

The foregoing results obtained by the different methods are fully as concordant as could be expected in a determination of this character. Attention is now called to the relative amounts of fat obtained by the different methods studied. Averaging all the results, we have the following:

	Per cent.
Provisional method	38.55
Provisional method modified	39.23
Extraction after drying	39.69
Babcock test.....	39.81

All the work done indicates consistently and conclusively that the present provisional method gives too low results. That this is due to incomplete extraction, we can easily show. Five portions of the same sample of cheese were extracted by the provisional method for fifteen hours. The residue was then powdered and the extraction continued for five hours. Below are the detailed results.

Per cent of fat obtained in 15 hours extraction.	Per cent of fat obtained by powdering and extracting 5 hours more.
(1) 38.07	(1) 0.36
(2) 38.37	(2) 0.27
(3) 38.41	(3) 0.24
(4) 38.31	(4) 0.46
(5) 38.61	(5) 0.18

The amount of fat obtained by powdering and further extracting the residue, varied from 0.18 to 0.46 per cent, and averaged 0.30 per cent. Even then the results were

lower than those obtained by any other method. The difference is probably due to some loss of fat in the process of grinding the cheese with copper sulphate at the start. It is difficult to avoid some loss in this operation. Hence, the present provisional method is subject to two sources of loss: First, loss in process of grinding cheese with copper sulphate; second, loss from incomplete extraction.

The provisional method modified gave on an average 0.68 per cent more fat than the provisional method. In this method the cheese is not ground until most of the fat has been extracted. The whole operation can be carried out somewhat more easily.

It will be seen that the amount of fat obtained by extraction after drying is still greater than by the provisional method or its modification. The fat thus obtained is dark brown in color, giving evidence of impurity. The reporter has not found time to ascertain the detailed reasons for this increased yield of extract, and can not therefore recommend this method at present. It is also noticed that the amount of fat obtained by the Babcock test is higher than by any other method, agreeing most closely with the results obtained by extracting the fat after drying. But this fat has not been examined as to its purity. If further work shall show that the extract which is highest in amount consists of equally pure fat, then it will be found desirable to use the method giving such results. On the basis of the data at hand, I can only recommend the substitution for the present provisional method of the modification which has been studied this year. I would suggest that further work be done in studying the purity of the fat obtained by extraction after drying cheese. The method which gives the largest amount of pure fat is the method desired.

DETERMINATION OF ACIDITY OF CHEESE.

An attempt was made to study the amount of total acids in cheese in accordance with the following instructions: Agitate 10 grams of cheese vigorously with nearly 100 cc of water at a temperature of 40° C.; filter; make up filtrate to 100 cc; titrate portions of 25 cc of the filtrate with a standardized solution of sodium hydroxid, preferably a tenth-normal solution. Use two different indicators for comparison—phenolphthalein and cochineal. Report the amount of actual sodium hydroxid used for 1 gram of cheese.

The sample of cheese used was too old to give satisfactory results for this work. The following suggestion was made by S. M. Babcock:

On account of the difficulty in filtering aqueous extracts of cheese, especially if the cheese is old, I would suggest that the extract be made up to 105 cc before filtering, 5 cc representing approximately the volume of the insoluble solids of the cheese when 10 grams are taken, and that 25 cc of the filtrate, representing 2.5 grams cheese, be titrated. This would save much time and, I believe, be fully as accurate as the method described, for it is impractical to completely wash the residue on the filter when only 100 cc of water are used.

RECOMMENDATIONS FOR CHANGES IN METHODS OF ANALYSIS OF CHEESE.

1a. *Preparation of sample.*

When the cheese can be cut, a narrow wedge-shaped segment reaching from the outer edge to the center of the cheese is taken. This is to be cut into strips and passed through a sausage-grinding machine three times, being mixed as completely as possible after each grinding, taking care to avoid evaporation of water. From the mass thus prepared samples are taken for analysis.

When the cheese can not be cut, samples are taken by a cheese trier. If only one plug can be obtained this should be taken perpendicularly to the surface at a point one-third of the distance from the edge to the center of the cheese. The plug should reach either entirely through or only halfway through the cheese. When possible draw three plugs, one from the center, one from a point near the outer edge, and one from a point halfway between the other two. For inspection purposes the rind may be rejected, but for investigations requiring the absolute amount of fat in the cheese the rind is included in the sample. It is preferable to grind the plugs

in a sausage machine, but when this is not done they are cut very fine and carefully mixed.

2b. *Determination of water.*

Change first sentence to read as follows: From 2 to 5 grams of the prepared sample of cheese are placed in a weighed porcelain or platinum dish which contains a small quantity of material, such as freshly ignited asbestos or sand, etc.

3c. *Determination of fat.*

Substitute for present method the modification described above in this report. Use 2 to 5 grams of material.

4. It is recommended that the following be adopted as a provisional method for the determination of total acids in cheese: Weigh 10 grams of cheese, add water at a temperature of 48° C. until the volume equals 105 cc, and agitate vigorously; filter; titrate portions of 25 cc of filtrate corresponding to 2.5 grams of cheese with a standardized solution of NaOH, preferably one-tenth normal. Use phenolphthalein for indicator. Express amount of acid as lactic acid.

5. It is recommended that the methods of cheese analysis as modified be made official except where otherwise provided.

6. It is recommended that the next reporter make a study of the fat of normal and filled cheese, in order to devise a method for identifying the latter.

7. It is recommended that the next reporter also consider the question of milk adulterants in form of preservatives and study methods of identification.

8. It is recommended to adopt the following as a provisional method for the determination of casein in cows' milk:

The determination of casein in milk should be made when the milk is fresh or nearly so. When it is not practicable to make this determination within twenty-four hours add 1 part of mercuric chlorid to 2,000 parts of milk and keep in a cool place. Weigh about 10 grams of milk, dilute in a beaker with about 90 cc of water at 40° to 42° C., and add at once 1.5 cc of a solution containing 10 per cent of acetic acid by weight. Stir with a glass rod and let stand three to five minutes or longer. Then decant on filter, wash two or three times with cold water by decantation, and transfer precipitate completely to filter. Wash once or twice on filter. The filtrate should be clear or very nearly so. If the filtrate is not clear when it first runs through it can generally be made so by two or three repeated filtrations, after which the washing of the precipitate can be completed. The washed precipitate and filter paper are then digested as in the regular Kjeldahl method for the determination of nitrogen, and the determination of nitrogen is completed as usual. To calculate the nitrogen into an equivalent amount of casein, multiply the per cent of nitrogen by 6.25.

In working with milk which has been kept with mercuric chlorid the acetic acid should be added in small portions, a few drops at a time, stirring after each addition, and continuing the addition of acetic acid until the liquid above the precipitate becomes clear or very nearly so.

9. It is recommended that the following be adopted as a provisional method for the determination of albumin in cows' milk.

The filtrate obtained above in separating casein is placed in a water bath and heated to the boiling temperature of water for ten or fifteen minutes. The filtered and washed precipitate is then treated by the Kjeldahl method for determining nitrogen. The amount of nitrogen multiplied by 6.25 gives the amount of albumin.

AMENDED AND ADOPTED REPORT OF THE COMMITTEE ON RECOMMENDATIONS IN RELATION TO DAIRY PRODUCTS.

It is recommended—

(1) That the method of sampling as recommended by the reporter be adopted as a substitute for the present method.

(2) That the amount of material (cheese) to be taken for water determination be changed to that recommended by the reporter.

(3) That the method recommended by the reporter be substituted for the present method under (c) *Ether extract*.

(4) That the method for the determination of total acid in cheese as described by the reporter be adopted as a provisional one.

(5) That the following methods on page 37 be made official, viz, (a), (b), (c), (d), (e), and (f), with the modifications as above given.

(6) That the method for casein in milk described in 1893 by Dr. Van Slyke be adopted as a provisional one.

(7) That the method for albumin in milk as described in 1894 be adopted as a provisional one.

(8) That the reporter for 1897 be authorized to study methods for identifying filled cheese.

(9) That the reporter for 1897 be authorized to study methods for the identification of milk adulterants used in the form of preservatives.

H. J. WHEELER,

For the Committee on Recommendations of Reporters.

A MODIFICATION OF THE BABCOCK METHOD AND APPARATUS FOR TESTING MILK AND CREAM.

By J. M. BARTLETT.

The modifications described in the paper are, briefly stated, as follows:

First. The modification of the method consists, chiefly, in filling the bottles with hot water after the milk or cream and acid are added, before they are put in the centrifugal machine and whirled, thereby completing the separation with one whirling and saving the time required for the second whirling.

Second. The modifications of the apparatus are: The base portion of the milk bottle is graduated, and the base portion of the cream bottle is reduced in size and graduated so no acid measure is required.

While working with the Babcock test some months ago it occurred to the writer that the hot water might be added directly after mixing the acid and milk together, and that separating the fat before adding water was an unnecessary part of the process, if the right conditions could be brought about. Accordingly, to decide the matter, a number of tests were made with all conditions the same, as in the regular method, except that hot water was added before the bottles were whirled. In this way only partial success was attained. A few of the bottles showed clear separations of fat, but many were cloudy, apparently containing curd. Success in few cases gave encouragement for further trials, so the investigation was continued until the following method was hit upon, which the writer has used successfully for the past six months with all kinds of milk and cream:

METHOD.

After the milk is mixed by stirring, or pouring from one vessel to another, the required amount, 17.6 cc, is measured into the test bottle. It is then heated to about 70° F., if not already at that temperature, by setting the bottle in a tank of warm water, 20 cc of sulphuric acid (sp. gr. 1.82 to 1.825) are added, and the bottle shaken by giving it a rotary motion until the milk and acid are thoroughly mixed. The mixture is then allowed to stand not less than five minutes. No harm is done if it stand longer, and, in fact, occasionally some kinds of milk have to be given a little more time. After standing the necessary time the bottle is given another gentle shake to mix in and dissolve any particles of curd that may have risen to the surface. Hot water is then added nearly to the 10 per cent mark, the bottle put in the cen-

trifugal machine, and whirled for five minutes at the rate of 1,000 to 1,200 revolutions per minute. A steam turbine machine is best for this purpose, but a hand or belt power machine can be used if hot water be put in the pan to keep the fat melted. After the whirling is completed the per cent of fat can be read off in the usual manner.

It would appear that the time required for the bottles to stand after the acid is mixed with the milk would offset that gained by omitting the second whirling, which is made in the old method; but the writer has often found it necessary with many kinds of milk, especially that from cows much advanced in the period of lactation, when the milk is very rich, to allow the bottles to stand a while when working the two-whirling method in order to get a clear separation of fat. However, everyone who does much testing should have at least two sets of bottles, so there would be no loss of time by this process. When two sets of bottles are at hand one set can be charged with the milk and acid and stand while the second set is filling, and the second set can stand while the first is whirling. It is much better to make twelve tests at a time than to make any larger number. Twelve are about all one can easily read off before the fat begins to cool and contract in volume.

For the above-described modification of the milk test no change of apparatus need be made, the writer, however, prefers to have the base portion of the bottle graduated as before mentioned, so no acid measure is required and only one pouring of the acid is necessary.

By having the bottle marked at a point at which it holds 37.5 cc, one can, after the milk is measured in with the pipette, run the acid in until it is filled to this point. It was found impracticable to use a bottle with a smaller base capacity, like the cream bottle described further on, because of the larger amount of curd in milk than in cream and the smaller size of the neck of the milk bottle necessitating more space for shaking, breaking up the curd, and dissolving it in the acid.

THE CREAM BOTTLE.

The bottle before mentioned and herein described is similar to the regular milk bottle, also the Connecticut station cream bottle, except that the base portion is made of such size as to avoid using an acid measure. The base is made to hold about 38 cc up to the neck, and after the cream is measured in the required amount of acid is added if the bottle is filled nearly to the neck. The neck portion is large enough to carry 25 per cent of fat, and is graduated to one-half per cent, but can be easily read to one-quarter per cent. Each per cent is numbered. Although one can not read as closely with this as with the bulb-neck bottle, which was designed to test both milk and cream, and described in Bulletin No. 3, Second Series, Maine Experiment Station, one can read with sufficient accuracy for all practical purposes. On account of the neck being larger and shorter the new style bottle is more easily cleaned than the old, is less liable to breakage, and by using the method previously given for milk a test can be made more rapidly. Twenty-five per cent was fixed upon as the capacity of the neck, for the reason that a much higher per cent necessitates an increase in the diameter, which impairs the accuracy in reading; and again, nearly all cream shipped to the creameries is raised by the cold deep-setting process, and will seldom contain more than 20 per cent of fat. If one wish to test separator cream that is very rich, 9 cc or 9 grams can be taken instead of 18, 9 cc of water added and the usual amount of acid. The per cent of fat obtained in that case of course should be multiplied by two to give a correct reading.

The method employed is practically the same as for milk. That good results and clear separations can be obtained by the previously described modifications the writer and others connected with the college have fully demonstrated. It is necessary, however, that the details of the method be strictly observed to attain success.

Below is given in tabular form a few of the many results obtained on milk and cream in comparing the old process with the one-whirling method:

Per cent of fat in milk.

Milk.		Milk.		Milk.		Cream.	
Whirled once.	Whirled twice.	Whirled once.	Whirled twice.	Whirled once.	Whirled twice.	Whirled once.	Whirled twice.
5.3	5.3	5.2	5.3	3.8	3.7	18 $\frac{3}{4}$	18 $\frac{3}{4}$
5.6	5.6	5.3	5.4	4.1	4.2	18 $\frac{1}{2}$	18 $\frac{3}{4}$
6.0	6.0	4.7	4.8	5.2	5.0	18 $\frac{3}{4}$	-----
4.9	4.9	5.0	4.9	5.1	5.2	18 $\frac{1}{2}$	-----
4.9	5.0	5.8	5.8	5.3	5.1	23 $\frac{1}{2}$	23 $\frac{1}{2}$
5.2	5.2	5.0	5.0	5.0	5.0	23 $\frac{3}{4}$	-----
4.6	4.6	4.9	4.9	5.0	5.2	17	17 $\frac{1}{4}$
5.0	5.1	4.9	5.0	5.7	5.8	19 $\frac{1}{2}$	19

The convention then adjourned till Monday morning.

THIRD DAY.

MONDAY—MORNING SESSION.

The PRESIDENT. We will now proceed with the report of the committee on recommendations of reporters.

Mr. WHEELER. There is one matter that I ought perhaps to have mentioned in connection with soils. If it be agreeable to the convention I will bring it up now; if not, will let it go until another time.

The PRESIDENT. I suppose there is no objection.

Mr. WHEELER. The point is this: We have performed experiments at some twelve points in Rhode Island and found that a number of soils are so deficient in bases that the reaction of the soil becomes acid, even on hilltops which have a gravelly subsoil where the drainage is excellent. It seems that the test for acidity may in some cases in upland and well-drained soils be very important as indicating a sufficiency of bases and it might be wise to have a test for the reaction of soil introduced into our soil analyses. It is recognized by many, but there are many others who do not take it into consideration. I presented a paper last year in this line. With us it is a most important matter.

The PRESIDENT. Any remarks on this subject?

Mr. WILEY. It is unusual for any of us to think of testing well-drained soil, but I think it is a matter of considerable importance and that it should be looked into more carefully hereafter. I think if the reporter were instructed to consider this question it would be a matter of importance. I move, therefore, that the reporter be instructed to consider the question of testing the acidity of soils.

Motion seconded and adopted.

Mr. WHEELER. I will now offer the recommendations on phosphoric acid, as agreed upon by the committee.

The recommendation that the volumetric method be adopted as official was the subject of the following discussion:

Mr. WILEY. The first problem which presents itself to my mind is one I supposed was settled by the national election—that is, Can we have two standards for the same thing? I believe that our standard method, which is recognized as a standard—that is, the gravimetric method—should remain as the only standard of value for the measuring of phosphoric acid, but I believe analysts should be allowed to use other methods in their routine work as well as in their official work. I would object, however, in the light of our present experience, to adopting it as an equal standard with the official gravimetric method. I believe we

would be taking a dangerous step in doing this, because the method is not yet sufficiently developed. It seems to me it would be a great deal better if we would allow it to remain as an optional method, but we ought not to try a double standard.

Mr. KILGORE. As to what is a standard, or what should be the standard, I do not think that any method should be taken as a standard, but a theoretical percentage of pure salt should be used, as in the case of the potash work. For the past three years the reporters rather than take any method as a standard have used the theoretical percentage of salt and by that method they have proven our present potash method to be a most excellent one. I take it that this pure phosphate salt should be taken as the standard rather than any one method. We ought to take what actually is as a standard rather than a method.

Mr. WILEY. How do we determine the purity of a salt? We would determine the purity of a phosphate salt by the gravimetric method. We can not rely upon any commercially or chemically pure salt which is sent out. We must first test it and be sure that the salt is pure. If the salt be certainly pure, as far as that particular salt is concerned the methods would be directly applicable. If we had the whole volumetric as an optional method or as an alternative method my opposition would at once disappear. But I would object as a member of this association to have it adopt the volumetric as a standard method or place it on an equality with the gravimetric method.

Mr. VAN SLYKE. As a member of the committee on recommendations, my understanding was that it was recommended as an optional method.

Mr. MYERS. I am disposed to take the position that Mr. Wiley has. I doubt the advisability of the association recognizing in full force a system of analyses which can not be worked uniformly by different chemists in different places, and which is treacherous under any circumstances, so that any chemist who may get the results correct a portion of the time may expect a disappointing result at an unexpected moment.

In regard to this volumetric method, we have tried honestly and very hard to get the chemists at our laboratory to use it so that we could employ it in our operations in fertilizer analyses. There are several reasons why it is desirable to have a quick method on fertilizer analyses if we can get a reliable one, but with four or five chemists I have had at work trying their best to get it into shape to work satisfactorily we have failed utterly to get results that we could depend on as we can upon the gravimetric method, and while the chemists at work have had full liberty to use it if they wished I notice they have uniformly abandoned it, for the reason that if they had used it they were very liable to have to review their work by the gravimetric method in order to determine whether their volumetric method was correct.

We have, it is true, an optional method in potash determinations, and I will venture to say that every chemist here, at least a large number of chemists, will agree with me that perhaps there is no element in fertilizer determinations at present that is in a more satisfactory condition than the potash determinations. We have a standard that is recognized the world over for phosphoric-acid determinations. Shall we stamp as official a method that will bring into that an element of confusion? I doubt very much the advisability of it. I have no objection to chemists, if they can use this method with precision in their laboratories, employing it if they wish to, but I doubt the advisability of adopting it as an alternative method. I think it should be published as a method to be employed at the option of the chemist if he fail in the other method. But I know it is impossible in some well-organized laboratories in this country to get concordant results by that method. I know that it is impossible for chemists of experience and most careful training to get concordant results by that method. There are elements of inaccuracy in it which have not yet been determined, and while my friend claims that a method giving concordant results upon pure salt has some elements of strength in the argument there are elements entering into this that weaken his argument, because he can not tell what he is working with when he has an impure salt in hand.

Mr. KILGORE. In about 325 results with the gravimetric method 276 samples of totals averaged by the official 10.70 per cent, by the volumetric 10.72 per cent. Fifteen solubles averaged by the official 2.61 per cent, by the volumetric 2.55 per cent. The results agreed very closely all the way through.

Mr. McDONNELL. I should like to see the association adopt it as an alternative method.

Mr. WHEELER. Would like to hear from Mr. Kilgore in regard to one point. The point has been raised that discordant results have been obtained in one laboratory. I would like to know if in the results reported from other laboratories such discordant results have been obtained or whether it is confined to this one laboratory.

Mr. KILGORE. I would merely refer you to the report of last year, where the method was worked in its present form (p. 76). I gave there, instead of averages, all the results. The results are very satisfactory upon all samples sent out. There were no discordant results that came into my hands last year.

Mr. JOHNSON. I wish to warn this association to proceed very cautiously. The volumetric method is extremely promising, but I am reminded of some of my own experiences in years gone—twenty years ago—when I sought to use one of the later methods as a shorter cut in fertilizer analysis, and at my instigation Mr. Wells, now Professor Wells, of Yale University, made an investigation of that method and he obtained a large number of results just as satisfactory as those obtained by the original method. But now and then we struck a snag, and

although for one entire fertilizer season every sample was analyzed by both methods, we finally had to give up the new method as inaccurate. We can not afford to make any mistakes; we can not afford to use a method that we do not positively know is the very best.

Mr. SCOVELL. Mr. Johnson has suggested a thing I have been thinking about for some time. If we adopt the volumetric as an alternative method, we adopt it as a legal method, because the courts have decided that official methods are legal methods. I remember a case this spring where a party got 2 per cent more phosphoric acid than we did. On investigation we found they were using the volumetric method, and I proved to them that that was not the official method, because we had not adopted it as an alternative method. If we adopt it as an alternative method these manufacturers could make these analyses by this method. That is, I think, a serious objection to adopting it as an alternative method. As the controller of fertilizers in my State I have to look further than most chemists. I have to sign all of the analyses, and I want to be sure before signing them, and I know by the present methods they are all right. I would hesitate if the analyses were made by the alternative method, because I might get into difficulty. If we can go before our courts and show that we are accurate, that we adopt only the best methods, we have won the battle already; but if we show that we adopt methods that are not accurate, the courts may decide that these are not the best methods. When we adopt a method we want it universal.

Mr. WHEELER. I will say that, for my part, I should hesitate very much to make this method a method to be exhibited in court under the present circumstances. I supposed that it was understood that in court cases the standard gravimetric method was the only method allowable.

Mr. HUSTON. I suppose it is understood that the committee in recommending this method act upon the light which is presented to the committee. We had three men at least who have done an enormous amount of work upon this, and they have spoken very favorably of this method. I am frank to state here, as I did before the committee, that I do not wish to prevent anyone who desires to use this method from using it, but I will not allow this method to be used in my own laboratory, because the results which we have obtained are very far from being satisfactory; at the same time, I do not consider I have given the matter a full trial. My assistant absolutely refused to do the work by that method. The class of material, however, which we have is different from the general fertilizer material. I feel, however, that our experience ought not to count against that of those who have used the ordinary material. It was not in our hands applicable to that kind of material. I think there are some present who are neither station men nor official chemists, and I hope these men will appreciate and make use of the invitation always accorded to make any remarks on this and other matters.

Mr. CARPENTER. I am not connected with the experiment stations now. As a rule, I think you will agree with me that the fertilizer men

try to put pure goods upon the market, and we want you to treat us in the same way. If this method can not be handled by all official chemists, it does not seem fair that it should be introduced—that is, if it can not be used as an official method it ought not to be used as an alternative method to be used by those disposed to do so. I myself have used the method and gotten good results. I think the great trouble has been in getting here and there a bad result. Now and then you will get a result 2 or 3 per cent higher or 2 or 3 per cent lower, and in either case it would work very badly. I think that is the experience of most workers with this method. While in general it gives good results, occasionally it gives bad results.

Mr. WHEELER. I would like to ask any others here who have used this method to state their experience.

Mr. RUNYAN. I have had considerable experience with the volumetric method in the last two years, and while the results compared very favorably with the gravimetric method, once in a while I would get a result that was very bad.

Mr. VAN SLYKE. From the evidence that was presented last year or even the year before, accompanied by the evidence presented this year by the reporter, I was of the opinion very strongly that there was scarcely any other method that had been subjected to such critical investigation which gave such satisfactory results; but since listening to the discussion this morning and finding out that there are exceptional cases coming when we least expect them, I am inclined to change my mind somewhat and coincide with the conservative position of the association, and I therefore move that we substitute for the recommendation of the committee the recommendation that this method be continued another year as a provisional method, and that the reporter for next year be asked to continue work on this method.

Mr. LINDSEY. I would suggest that there be inserted “upon a variety of samples.”

Motion as amended to include substitute adopted.

Mr. LINDSEY. I think every station ought to give this volumetric method a very careful trial.

Mr. WHEELER. I would like to say that when the report on phosphoric acid was made there was no objection made to this method. I would say also that the committee has to act just as a jury—upon the evidence presented. The facts presented were all favorable to this method, and hence our recommendation.

The other recommendations of the committee were adopted without discussion or division, and the final form of the recommendations and changes as adopted follow.

Mr. HUSTON. I think it would be the part of wisdom on the part of this association to instruct its reporter to take into consideration the matter of methods of determination of total and so-called available phosphoric acid in basic slag.

Mr. MYERS. There will undoubtedly be a demand for that during the coming year. Every station east of the Mississippi and throughout the Eastern and Southern country will be confronted by problems necessitating something upon that subject. I think the reporter at least should take hold of it during the coming year. Everything seems to be in chaotic condition. For my part I do not know what should be done with it. I move it be referred to the reporter on phosphoric acid for report at the next meeting.

Adopted.

The PRESIDENT. The recommendations adopted by the committee on soil analysis will now be presented.

Mr. Wheeler presented the report of the committee, which was adopted. The recommendations and changes are as given in the report, which is found immediately after the report on soil analysis.

Mr. VAN SLYKE. There was no opportunity given to read papers on dairy products, and I have a paper by Mr. Bartlett.

The PRESIDENT. That will now be in order.

(The paper is found immediately following the "Report on dairy products.")

The PRESIDENT. The next thing in order is the report on sugar. I received a letter from Dr. Nicholson, the reporter on this subject, several months ago, stating that he understood the meeting would take place earlier in the summer, and he was not aware he had been appointed reporter, and that he was unable to prepare any report.

The next report in order will be that of fermented and distilled liquors.

Mr. BIGELOW. I regret very much that I have practically nothing to report this year. Samples were sent to 17 chemists, who requested them, with directions for the estimation of glycerol and fusel oil. Not a single report was received, however, and my own results would not warrant the change of any of the methods which have been in use. I have several additions, however, to recommend as provisional methods for the examination of distilled liquors.

PROVISIONAL METHODS FOR THE EXAMINATION OF DISTILLED LIQUORS.

DETERMINATION OF FUSEL OIL.

The apparatus recommended for this determination is Bromwell's modification of Roese's fusel-oil apparatus.

The reagents required are fusel-free alcohol that has been prepared by fractional distillation over caustic soda or caustic potash, rejecting the first and the last three-fifths of the distillate, and diluted to exactly 30 per cent by volume (sp. gr. 0.96541), chloroform, freed from water and redistilled, and sulphuric acid (sp. gr. 1.2857 at 15.6°).

Distill slowly 200 cc of the sample under examination till about 175 cc have passed over, allow the distilling flask to cool, add 25 cc of water, and distill again till the total distillate measures 200 cc. Dilute the distillate to exactly 30 per cent by volume¹ (sp. gr. 0.96541 at 15.6°).

Now prepare a water bath, the contents of which are kept at exactly 15°, and place in it the apparatus (covering the end of the tube with a rubber cap to prevent wetting the inside of the tube) and flasks containing the 30 per cent fusel-free alcohol, chloroform, sulphuric acid, and the distillate diluted to 30 per cent by volume. When the solutions have all attained the temperature of 15°, fill the apparatus to the 20 cc mark with chloroform, drawing it through the lower tube by means of suction, add 100 cc of the 30 per cent fusel-free alcohol and 1 cc of the sulphuric acid, invert the apparatus and shake vigorously for two or three minutes, interrupting once or twice to open the stopcock for the purpose of equalizing pressure. Allow the apparatus to stand ten or fifteen minutes in water that is kept at the temperature of 15°, turning occasionally to hasten the settling of the chloroform, and note the volume of the chloroform. After thoroughly cleansing and drying the apparatus repeat this operation, using the diluted distillate from the sample under examination in place of the fusel-free alcohol. The increase in the chloroform volume with the sample under examination over that with the fusel-free alcohol is due to fusel oil, and this difference (expressed in cubic centimeters) multiplied by the factor 0.663 gives the volume of fusel oil in 100 cc, which is equal to the percentage of fusel oil by volume in the 30 per cent distillate. This must be calculated to the percentage of fusel oil by volume in the original liquor.

Example.—A sample of liquor contains 50 per cent of alcohol by volume. The increase in the chloroform volume with the 30 per cent fusel-free alcohol is 1.42 cc. The increase in the chloroform volume with the distillate from the liquor under examination diluted to 30 per cent is 1.62 cc; difference, 0.20 cc. The volume of

¹The following is an accurate method for diluting any given alcohol solution to a weaker solution of definite percentage: Designate the volume percentage of the stronger alcohol by V and that of the weaker alcohol by v . Mix v volumes of the stronger alcohol with water to make V volumes of the product. Allow the mixture to stand till full contraction has taken place and till it has reached the temperature of the original alcohol and water and make up any deficiency in the V volumes with water.

Example.—It is desired to dilute a distillate containing 50 per cent of alcohol by volume until it contains 30 per cent. To 30 volumes of the 50 per cent alcohol add enough water to make 50 volumes, or place 150 cc of the distillate in a 250 cc flask, fill to the mark with water, mix, cool, and fill to the mark again.

fusel oil in 100 cc of the 30 per cent distillate, then, is $0.20 \times 0.663 = 0.1326$, and by the proportion $30:50::0.1326:0.221$ we obtain the percentage of fusel oil by volume in the original liquor.

DETERMINATION OF ALDEHYDS.

(a) Preparation of reagent.

Eighty cubic centimeters of a saturated solution of sodium bisulphite are mixed with a solution of 0.12 gram of fuchsin in about 800 cc of water, 12 cc of sulphuric acid added, the solution thoroughly mixed and diluted with water to 1 liter.

(b) Determination.

A portion of the sample is diluted with water or strengthened with aldehyd-free alcohol until it contains 50 per cent of alcohol by volume, and 25 cc of this solution are treated with 10 cc of the reagent and allowed to stand twenty minutes. At the same time 25 cc of a solution of 0.05 gram of acetic aldehyd in 1,000 cc of 50 per cent alcohol are treated in the same manner and allowed to stand the same length of time. The relative intensity of the colors of the two solutions is then determined by means of a colorimeter, and from the figure thus obtained the weight of aldehyd is estimated as acetic aldehyd, and calculated to percentage of the original liquor.

DETERMINATION OF ETHEREAL SALTS.

After the determination of the volatile acids the neutralized distillate is transferred to a flask connected with a reflux condenser, treated with 25 cc of tenth normal sodium hydroxid, and boiled one-half hour. The flask and contents are then cooled, 25 cc of tenth normal hydrochloric acid added, and the excess of acid titrated with sodium hydroxid, using phenolphthalein as indicator. The number of cubic centimeters of tenth normal alkali used in this titration, multiplied by 0.0088, is equal to the weight in grams of ethereal salts (calculated as ethyl acetate) in the volume of liquor taken for the determination.

It might be of interest at this point to notice the apparatus that is most commonly used for the determination of fusel oil.

The first method to be proposed for the estimation of fusel oil in alcoholic beverages which is worthy of serious attention was that of Roese.¹ He made use of the apparatus you see here (fig. 1). It consists of a stoppered cylinder graduated to 0.2 cc from 20 cc to 45 cc and is enlarged to some extent above the 45 cc mark. The entire capacity of the cylinder is from 175 to 200 cc. In the determination the apparatus is filled to the 20 cc mark with chloroform, 100 cc of the sample (diluted with water, or strengthened with fusel-free alcohol, until it contains exactly 50 per cent of alcohol by volume) added, the two

liquids thoroughly mixed by agitation, the chloroform allowed to subside and its volume noted. Throughout the operation the temperature of 15° must be maintained. A blank experiment is also conducted, using 100 cc of 50 per cent fusel-free alcohol instead of the diluted sample. The volume of the fusel oil present is equal to about 66.3 per cent of the increase of the chloroform volume with the diluted sample, less that with the fusel-free alcohol.

This apparatus and the method of its application have been modified by Stutzer and Reitmair,² Herzfeld,³ and Windisch⁴ until now 30 per cent alcohol are employed

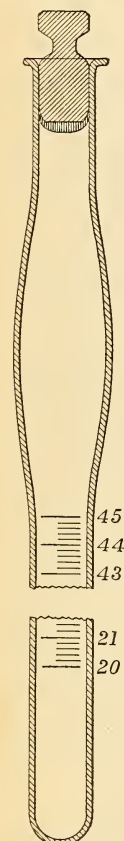


FIG. 1.—Roese's fusel-oil apparatus.

¹ B. Roese, *Ztschr. Spiritusind.*, 1884, No. 43.

² A. Stutzer und O. Reitmair, *Rep. anal. Chem.*, 1886, 6, 335-341, and 385-391.

³ H. Herzfeld, *Chem. Ztg.*, 1888, 12, 410.

⁴ C. Windisch, *Arb. a. d. kais. Ges. Amt.*, 5, 373-393.

instead of 50 per cent, 1 cc of dilute sulphuric acid is added to assist in the separation of the chloroform, and the apparatus itself has assumed the shape you see here (fig. 2). We find here that the graduated portion of the apparatus is made so much smaller in diameter that the error in reading is reduced to 0.01 cc. This form of apparatus, however, is open to two serious objections.

First. It is very difficult to fill it exactly to the 20 cc mark without touching the sides of the tube and thus adding an excessive amount—and, indeed, an unknown amount—of chloroform.

Second. The smallness of the stem makes the cleansing of the apparatus slow and inconvenient.

To obviate these objections a further modification has been suggested by Bromwell¹ (fig. 3). This consists in sealing a stopcock tube to the bottom of the chloroform bulb, to facilitate the

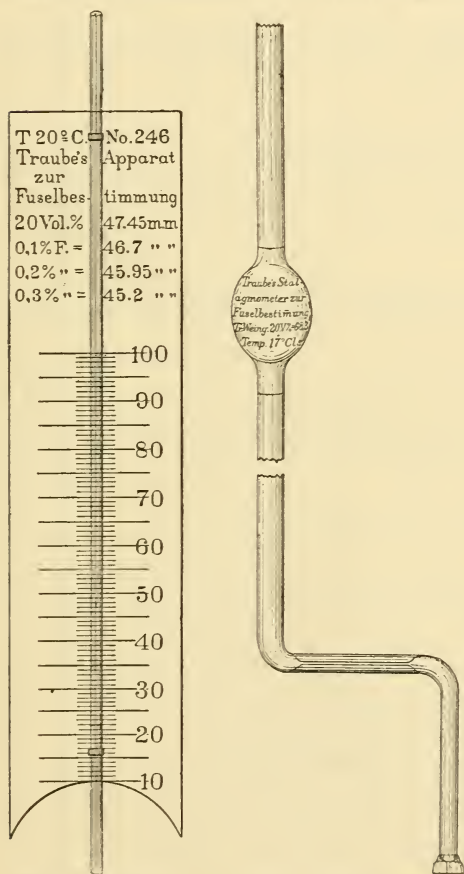


FIG. 4.—Traube's capillarimeter.

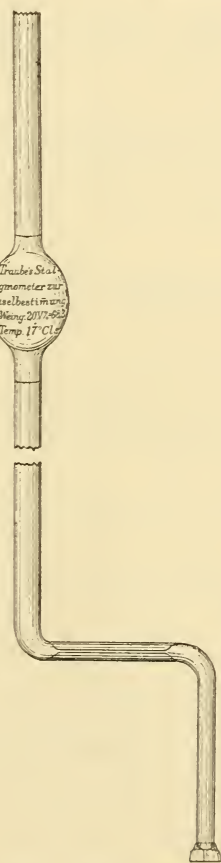


FIG. 5.—Traube's stalagmometer.

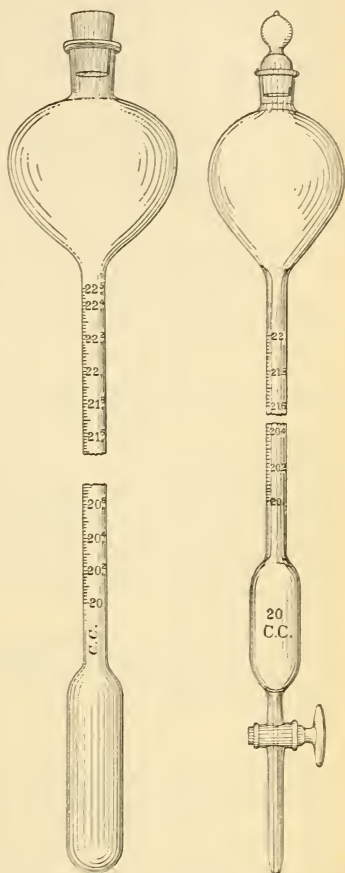


FIG. 2.—Stutzer and Reitmaier's fusel-oil apparatus.



FIG. 3.—Bromwell's fusel-oil apparatus.

introduction of the chloroform and the cleansing of the apparatus.

The other apparatus to which I wish to call your attention are Traube's capillarimeter and stalagmometer, whose application depends on the influence of fusel oil on the viscosity of alcoholic liquors. The advantages claimed for the forms of apparatus are the convenience and rapidity with which they may be used. In each case the alcohol is diluted to 20 per cent by volume.

The first of these, in point of time as well as in point of value, is the capillarimeter (fig. 4). This consists of a capillary tube mounted on a porcelain scale. The scale

¹ William Bromwell, Bull. 47, p. 100.

is held in position by a clamp that may be raised and lowered by a screw until these two points (a and b) just touch the surface of the sample (diluted to 20 per cent by volume), which is contained in a dish or beaker below the apparatus. The sample is drawn to the top of the capillary tube several times by means of suction, and the height of the liquid in the tube is read on the porcelain scale. This reading is corrected for temperature, if not made at the temperature for which the instrument was graduated, and subtracted from the height of the column with 20 per cent fusel-free alcohol, which gives a figure from which the volume of fusel oil may readily be calculated by means of data given on each instrument.

The other apparatus of Traube's to which I referred is the stalagmometer (fig. 5). This consists of a bent bulb tube with the horizontal section and the point capillary. It is filled with the sample under examination (diluted to 20 per cent by volume), the number of drops between the two points of graduation above and below the bulb being counted. In a volume of the diluted sample, equal to 100 drops of 20 per cent fusel-free alcohol, each 1.6 drops in excess of that number indicates the presence of 0.1 per cent of fusel oil. The temperature for which the apparatus is graduated and a temperature correction are stated on each instrument.

AMENDED AND ADOPTED REPORT OF THE COMMITTEE ON RECOMMENDATIONS IN RELATION TO LIQUORS.

It is recommended—

(1) That the Röse method for fusel oil as modified by Stutzer and Reitnair, with Brownell's apparatus, be adopted as a provisional method.

(2) That the methods described by the reporter for aldehyds and esters be adopted as provisional ones.

(3) That any further work in the line of liquors be discontinued until called for by the association.

(4) That methods for detecting the adulteration of foods, so far as such are not provided for in connection with other reporters, be referred to the reporter on spirituous liquors.

H. J. WHEELER,

For the Committee on Recommendations of Reporters.

Mr. WINTON. It appears that this reporter on liquors has had an easy time of it, although he has distributed bottles, but no one seems to have had any time for analytical work. I do not think he has had his share of the work. It is very evident that a work of that nature is carried on to a great extent. There hardly seems to be work enough for the reporter. As I understand it, this examination of liquors pertains largely to the detection of adulterants. We know that the Department of Agriculture for a number of years past has done valuable work in examining foods for adulterants. Their publications are regarded as valuable works on this subject. At some of the agricultural stations work in this line has been carried on. It seems to me very proper that we should have a reporter for all this. I would therefore move that the duty of the reporter on fermented and distilled liquors be increased, and that he may be made the reporter of methods for the detection of adulterants in food. Of course a great deal of that work is carried on by other reporters. The reporter on dairy products considers the matter of detecting adulterants in milk, cheese, and butter, etc., but there is no need of their work interfering.

Mr. HIRD. In regard to the recommendation of the use of this method of determining fusel oil: There is perhaps at the most in the samples

of liquors but two to three tenths of 1 per cent, so that the use of the apparatus requires considerable care. Where the method is accurately carried out and the specific gravity is taken, as it should be, the method assures great accuracy. In all cases where this method is used, I might state that if carried out as it should be it insures great accuracy, and that if the operator in making use of the same fails to secure the results that should be secured he must not blame the method.

The **PRESIDENT**. The question arises whether this matter of increasing the duty of the reporter on liquors is proper.

Mr. **LINDSEY**. I do not know how we could get out of the matter, unless we refer it to the committee on recommendations.

Mr. **MYERS**. I move that the matter be referred to the committee on recommendations.

Mr. **FREAR**. I would like to say regarding food adulterations that I have had a little work to do in that line. I wish we had had a reporter on the subject five years ago. I would be heartily glad if the association in some form would provide for beginning work in this direction. It seems to me a proper subject for the association to undertake, since it is a matter of more than ordinary importance.

Mr. **MYERS**. I hope the motion to get it into some kind of shape will go forward. I am perfectly willing, however, to accept anything else, so we get it into proper shape.

The **PRESIDENT**. Recommendations are referred in regular order, and it seems to me this would be referred without any direct motion. If there is no objection, the recommendations are referred to the committee.

Mr. **MYERS**. It is now half past 12, and I think we had better take a recess until 2 o'clock. I move, therefore, that we take a recess until 2 o'clock.

Objection to this motion, and point raised that it would be better to stay in session and finish proceedings.

Motion defeated.

The **PRESIDENT**. The report on tanning materials is now in order.

Reporter not present.

Mr. **YOCUM**. In the absence of the reporter I may be allowed to say that we feel deeply indebted to this association. The publications of the association have been sent around among the tanners and extract manufacturers, and if to-day an analysis is made the extract man is not satisfied unless it is the method printed in the association's records; neither is the tanner. I can not go further than that, beyond saying we buy a good deal of extract, and it amounts to considerable, looking at it entirely from a commercial point of view. I feel greatly interested in that line from that point of view, and am deeply indebted to this association for having brought it into the position where tanner and extract man have got to come down and use these analyses as the basis on which the extracts have to be sold and bought. Seventy-five per

cent of the extract bought is paid for on the analysis, and the analysis is made by the official methods.

Messrs. Krug, Yocum, Haley, and Carr presented recommendations in respect of tanning methods, which were referred to committee on recommendations.

The committee recommended the adoption of the proposed changes.

Mr. WILEY. I move that the report of the committee on recommendations made on tanning be accepted as the action of the association. Adopted.

The recommendations as adopted follow:

AMENDED AND ADOPTED REPORT OF THE COMMITTEE ON RECOMMENDATIONS IN RELATION TO METHODS FOR THE ANALYSIS OF TANNING MATERIALS.

(1) *Preparation of sample.*

Barks, woods, leaves, and similar tanning materials should be ground to such a degree of fineness that they can be thoroughly extracted.

Extracts must be heated to 50° C., well shaken, and allowed to cool slowly to room temperature.

(2) *Quantity of tanning material.*

In the case of barks, woods, leaves, etc., use such an amount of the material as to give about one gram of total solids per 100 cc of solution, and extract in a Soxhlet or similar apparatus. In the case of extracts, weigh 20 grams dilute with water at 80°, cool slowly to 20°, and make up to a liter.

(3) *Determination of moisture.*

Dry 5 grams of the sample in a flat-bottomed dish at the temperature of boiling water until the weight becomes constant.

(4) *Determination of total solids.*

Shake the solution and without filtering immediately measure out 100 cc with a pipette. Care must be taken to keep the solution at 20°. Evaporate in a weighed dish and dry to constant weight at the temperature of boiling water. The dishes in which the drying is conducted should be covered while they are in the oven in order to prevent loss.

(5) *Determination of soluble solids.*

Filter about 125 cc of the solution (at 20°) through a double-folded filter (S. and S. No. 590, 25 cm), returning the filtrate through the filter twice. Evaporate 100 cc as before.

(6) *Determination of tanning substances.*

Prepare 20 grams of hide powder by shaking for five minutes with 250 cc of water and straining through linen. Repeat the operation three times. Finally remove as much water as possible by squeezing in a press. Transfer the pressed hide powder to a covered dish and weigh. Dry to constant weight, at 100°, a portion approximately equal to one-fourth of the whole. To 200 cc of the original unfiltered solution (at 20°), add the remainder of the hide powder, and shake for ten minutes. Add 5 grams of barium sulphate free from soluble salts and shake again for one minute. Filter immediately through a folded filter (S. and S. No. 590, 15 cm), returning the first 25 cc. Evaporate 100 cc as before. The weight of the residue must be corrected for the dilution caused by the water contained in the hide powder. The shaking must be done in some form of mechanical shaker. The simple machine used by druggists, and known as the milk shake, is recommended.

(7) *Testing the hide powder.*

(a) Shake 10 grams of the hide powder with 200 cc of water for five minutes; filter through muslin or linen; squeeze out thoroughly by hand; collect the hide powder, and repeat the operation twice with the same quantity of water. Pass the last filtrate through paper until a perfectly clear liquid is obtained. Evaporate 100 cc

of the final filtrate in a weighed dish; dry at 100° until the weight is constant. If the residue amount to more than 10 milligrams the sample should be rejected. The hide powder must be kept in a dry place and tested once a month.

(b) Prepare a solution of pure gallotannic acid by dissolving 5 grams in 1 liter of water. Determine the total solids by evaporating 100 cc of this solution and drying to constant weight. Treat 200 cc of the solution with hide powder exactly as described in paragraph 6. The hide powder must absorb at least 95 per cent of the total solids present. The gallotannic acid used must be completely soluble in water, alcohol, acetone, and acetic ether, and should contain not more than 1 per cent of substances not removed by digesting with excess of yellow mercuric oxid on the steam bath for two hours.

(8) *Testing the nontannin filtrate.*

(a) For tannin: Test a small portion of the clear nontannin filtrate with a few drops of a 10 per cent solution of gelatine (Nelson's). A cloudiness indicates the presence of tannin, in which case the determination must be repeated, using 25 grams of hide powder instead of 20 grams.

(b) For soluble hide: To a small portion of the clear nontannin filtrate add a few drops of the original solution, previously filtered to remove reds. A cloudiness indicates the presence of soluble hide due to incomplete washing of the hide powder. In this case repeat the determination with perfectly washed hide powder.

Mr. Frear, the president elect, announced the committees and reporters for next year. The names of the members designated to act will be found in the list of officers. The president called for the report of the committee on pure-food legislation.

Mr. WILEY. I am sorry the committee has no formal report to make, and the fault is entirely mine. Owing to absence I neglected to bring the matter definitely before the other members of the committee, and the result is that no action has been taken. I beg the indulgence of the association for not having carried out its will, and ask that the committee be continued for another year.

The request for the continuation of the committee was granted.

The president called for the report of the committee on abstracts.

The committee presented the following report:

REPORT OF THE COMMITTEE ON ABSTRACTS.

MR. PRESIDENT AND MEMBERS OF THE ASSOCIATION: Your committee has the honor to make the following report. As soon as practicable after the appointment of the committee the work was organized and gotten under way, and the following assignment of work made:

E. W. Allen: Chem. Centralbl., Chem. Ztg., Ann. Chim. et Phys., Bull. Soc. Chim. Paris, Forsch. Geb. agr. Phys.; and the abstract part of Jour. Chem. Soc., Analyst, Ztschr. anal. Chem., Ztschr. angew. Chem., and Ber. deut. chem. Ges.

J. L. Beeson: Work on sugar analysis published in the French language.

W. D. Bigelow: Methods for fermented and distilled liquors.

B. W. Kilgore: Analyst, Chem. News, and Ztschr. anal. Chem.

W. H. Krug: Work on sugar analysis published in the German language, methods for tannin; and Amer. Jour. Sci., Amer. Jour. Pharm., Jour. Franklin Inst., and Pharm. Jour.

C. L. Parsons: Ztschr. anorgan. Chem., Ztschr. angew. Chem., and Amer. Chem. Jour.

H. J. Patterson: Jour. Amer. Chem. Soc., Jour. Chem. Soc. (London), Sci. News, and Boston Jour. Chem.

A. M. Peter: Jour. Soc. Chem. Ind., Ber. deut. chem. Ges., and Liebig's Ann. Phys. und Chem.

F. W. Woll: Landw. Vers.-Stat., Ztschr. Nahr.-Untersuch. und Hyg., Ztschr. physiol. Chem., and Milch-Ztg.

The limited number of journals within the reach of some of the members rendered it impossible to make an entirely equal division of the work. An effort was made, as far as the journals at the disposal of the committee permitted, to make an assignment by subjects.

With the exception of those to whom subjects were assigned, the abstracts were confined to the original articles appearing in their journals, omitting any abstracts which might be included in these journals. It was urged that the abstracts be sent in regularly and as promptly as possible, as the Office of Experiment Stations would cooperate with the association and would undertake to a considerable extent to follow up the principal abstract journals and otherwise supplement the work which the committee might be unable to do.

In the interest of uniformity the following suggestions were sent out by the chairman:

SUGGESTIONS FOR COMMITTEE ON ABSTRACTS, 1895-96.

The abstract should state (1) the English translation of the title; (2) the author; (3) the reference; (4) a concise summary of the contents of the article, which should be full enough to bring out the main points; and (5) the name of the abstractor.

In giving the reference, state the name of the periodical (using full and intelligible abbreviations, if preferred), the volume number in figures, the year, the number within the volume (No., heft, fasc., etc.), and the pages (beginning and ending of article), with the number of figures. The abstracts of chemical methods in Experiment Station Record will serve for illustration.

Govern the length of the abstract by the importance and value of the article, as far as practicable. Articles not deemed worthy of abstracting would be listed by title, thus: "A new method for the quantitative determination of glucose, F. GAND (Rev. Internat. Falsif., 8 (1895), No. 10, p. 173)." Usually a few words of explanation of the character of the article may be added with advantage.

When the abstract is made from an abstract instead of from the original article, give reference to both the original and the abstract, thus: (Chem. Ztg., 18 (1894), No. 58, pp. 601-603; abs. in Chem. Centbl., 1894, II, No. 3, p. 105).

Either abstract or list everything bearing on methods or apparatus in the journals assigned to you. This is very important, in order to make our work complete as far as it goes.

Exceptions.—(1) Where journals containing abstracts have been assigned to you, confine yourself to the original papers, as the abstracts have been provided for otherwise.

(2) Do not abstract or list methods for sugar, tannin, and fermented and distilled liquors, as these will be looked after by Messrs. Beeson, Krug, and Bigelow.

The work of the committee began with the issues of journals subsequent to September 1, 1895, where it was discontinued by the preceding committee. The publication of the abstracts began in Experiment Station Record, Vol. VII, No. 5, and has continued through each number up to Vol. VIII, No. 3, now in press. The printed work of the committee has occupied over 75 pages in the Record. There remain a considerable number of abstracts received since the last Record went to press, which will be included in the next number. It is believed that the work, in most lines at least, is well up to date.

The committee desires to acknowledge the efficient assistance rendered by Mr. W. H. Beal, of the Office of Experiment Stations, who has contributed many abstracts on fertilizers and soils.

E. W. ALLEN,
J. L. BEESON,
W. D. BIGELOW,
B. W. KILGORE,
W. H. KRUG,
H. J. PATTERSON,
W. L. PARSONS,
A. M. PETER,
F. W. WOLL,

Committee.

The president called for the report of the committee on resolutions. The committee presented the following report:

Resolved, That this association has heard with great sorrow of the death of Hon. Edwin Willits, late Assistant Secretary of Agriculture; that during the time Mr. Willits was Assistant Secretary we found him a true and helpful friend of our association, ready at all times to assist us with word and deed. After he returned to private life he did not lose his interest in our welfare, nor did he fail to attend our meetings. We remember with special pleasure his presence at our last meeting, and the words of cheer and advice which he gave us. His manly life and devotion to duty will ever be to us a support and inspiration to greater endeavor.

That a copy of this resolution be printed as a part of our proceedings, and that one be sent to the family of our dead friend.

B. W. KILGORE.

H. A. HUSTON.

H. W. WILEY.

Mr. MYERS. I move that the association extend to the Secretary of Agriculture our most hearty thanks for the courtesy he has extended us in publishing our reports and in providing facilities for conducting our meetings.

Mr. HUSTON. I think we should adopt a resolution of thanks to the National Museum.

The PRESIDENT. Both resolutions will be presented together. Both are unanimously adopted.

Calls for a speech from the president.

The PRESIDENT. I am very thankful for the very kind and courteous manner in which the presiding officer has been treated by all the members of the association.

Motion to adjourn *sine die* carried.

OFFICERS, REPORTERS, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1897.

PRESIDENT.

WILLIAM FREAR, State College, Pa.

VICE-PRESIDENT.

A. L. WINTON, Washington, D. C.

SECRETARY.

H. W. WILEY, Washington, D. C.

ADDITIONAL MEMBERS OF EXECUTIVE COMMITTEE.

Mr. B. W. KILGORE, Raleigh, N. C.

Mr. ARTHUR GOSS, Mesilla Park, N. Mex.

REPORTERS.

Phosphoric acid.—Mr. H. B. McDonnell, College Park, Md.

Nitrogen.—Mr. J. P. Street, New Brunswick, N. J.

Potash.—Mr. A. L. Winton, New Haven, Conn.

Soils and ash.—Mr. A. Goss, Mesilla Park, N. Mex.

Dairy products.—Mr. L. L. Van Slyke, Geneva, N. Y.

Foods and feeding stuffs.—Mr. J. B. Lindsey, Amherst, Mass.

Fermented and distilled liquors.—Mr. W. D. Bigelow, Washington, D. C.

Sugar.—Mr. L. W. Wilkinson, New Orleans, La.

Tannin.—Mr. B. D. Westenfelder, Cincinnati, Ohio.

ASSOCIATE REPORTERS.

Phosphoric acid.—Mr. L. H. Merrill, Orono, Me.

Nitrogen.—Mr. R. J. Davidson, Blacksburg, Va.

Potash.—Mr. C. H. Jones, Burlington, Vt.

Soils and ash.—Mr. Harry Snyder, St. Anthony Park, Minn.

Dairy products.—Mr. C. L. Penny, Newark, Del.

Foods and feeding stuffs.—Mr. F. W. Morse, Durham, N. H.

Fermented and distilled liquors.—W. S. Sweetser, State College, Pa.

Sugar.—Mr. Samuel Avery, Lincoln, Nebr.

Tannin.—Mr. John H. Yocum, New York, N. Y.

ABSTRACT COMMITTEE.

Mr. E. W. Allen, Washington, D. C., chairman.

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Mr. J. T. Anderson, Auburn, Ala.

Mr. B. H. Hite, Morgantown, W. Va.

Mr. W. H. Krug, Washington, D. C.

Mr. B. W. Kilgore, Raleigh, N. C.

Mr. F. W. Woll, Madison, Wis.

COMMITTEE ON PURE FOOD LEGISLATION.

Mr. H. W. Wiley, Washington, D. C., chairman.

Mr. H. A. Huston, Lafayette, Ind.

Mr. John A. Myers, Morgantown, W. Va.

Mr. A. S. Mitchell, Milwaukee, Wis.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy of the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members, and to have all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the president, at the regular annual meeting, a reporter and associate reporter for each of the subjects to be considered by the association.

It shall be the duty of these reporters to prepare and distribute samples and standard reagents to members of the association and others desiring the same: to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of fertilizer analyses, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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